ADAMA SCIENCE AND TECHNOLOGY UNIVERSITY
School of Mechanical, Chemical and Materials Engineering
Department of Materials Science and Engineering

Title: **Synthesis and Characterizations of Composite Material from a Clay Incorporated Polyethylene Terephthalate (PET)**

Researcher:-Tesfay G/Michael (MSc)
Co. researcher: Dr. NRR Anbusagar

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Abbreviation

ASTM	American Society for Testing and Materials
CNT	Carbon Nano Tube
CTE	coefficient of thermal expansion
EDS	Energy dispersive X-ray spectroscopy
GF	Glass fiber
HDPE	High density polyethylene
MAPP	Maleated polypropylene
MSW	Municipal Solid Waste
NC	nanoclay
OMMT	Preparation of Organic Montmorillonite
PET	Polyethylene Terephthalate
PP	Polypropylene
PVC	Polyvinyl Chloride
RPET	Recycled polyethylene terephthalate
SEM	Scanning electron microscopy
UP	Unsaturated Polyester Resin
UTM	Universal Testing Machine,
WPC	Wood plastic composites
XRD	X-ray diffraction

Abstract

Management of solid waste is an important problem in today's world. People pursue higher quality of life as a result of economic growth and development. Plastics have become an integral part of our lives. Its low density, strength, user- friendly design and fabrication capabilities, and low cost are the drivers to such growth. Hence, we indicate the management of solid waste and producing new materials from spring water bottle, which can be used for various applications. The recycling of Polyethylene Terephthalate (PET), which is a major post-consumer contributor of Municipal Solid Waste (MSW), could be used to produce a new dielectric material by incorporated with Na^+ *Montmorillonite clay. After collecting the PET samples from the MSW the recycling of PET would be started. The recycled PET is to be used as the matrix to manufacture the nanocomposites. In this Project Montmorillonite Nano-clays is used as fillers in different weight ratios by melt compounding method by using injection molding machine. The resultant melt compounded PET nanocomposites (available in the form of Pellets), which are to be tested for their mechanical, morphological and electrical properties as per ASTM standard specimen. It is found that material properties such tensile, flexural dielectric strength, dielectric constant and volume resistivity are improved at 4% clay loading. Further, it is noticed materials such as impact strength and hardness are improved at 6% clay loading.*

Keywords: Solid waste, Composite, Clay, Polyethylene Terephthalate, Nanomaterial

Chapter I: Background of the study

1.1 Introduction

Interests of scholars, academicians and industrialists are increasing on the previous years on exploration, investigating and producing of composite materials. According to Stojanović (2013) and Džunić (2015) explanations, composite materials are produced by adding basic material of the reinforcement in order to improve certain mechanical, physical, chemical and other characteristics, depending on the purpose of the selected base material and the reinforcement. According to Sandra *et al* (2018) explanation, composites can be classified in into three these compositions are: Metal base composite (MMC-Metal Matrix Composite), Polymer matrix composites (PMC-Polymer Matrix Composite) and a ceramic matrix when it is about composites with ceramic matrix (CMC-Ceramic Matrix Composite).

If those composite materials are nanoscale their application is attractive and promising at large. That's why Nano science and technology are among the prominent research areas of 21 century. As stated by Charles (2013), dimensionality plays a critical role in determining the properties of matter and the term "nanotechnology" can be defined as the controlled manipulation of materials with at least one dimension less than 100 nm. This field or Nanotechnology is totally restructure the technological applications in the fields of semiconductors, inorganic, as well as organic materials, energy storage, and biotechnology.

According to Charles (2013), Nanocomposites can dramatically improve properties like: · Mechanical properties including strength, modulus and dimensional stability; Electrical conductivity; Decreased gas, water and hydrocarbon permeability; · Flame retardancy; Thermal stability and Chemical resistance. Moreover, according to May-Pat *et al (2011),* Nanocomposite materials often possess a combination of physical properties that are not present in conventional polymer matrix composites. Because of their high aspect ratios, adding low concentrations of carbon Nano -tubes (CNTs) into a polymer matrix can improve the mechanical, thermomechanical and electrical properties of these polymer composite materials

Due to the above and many reasons, Nano reinforcement of polymers to form nanocomposites have attracted the attention of researchers for their potential in property development. Organically, unmodified and modified Nano-clay, Nano-fiber or carbon nanotubes having ultra-high strength and stiffness have become very popular during the last decade for Nano reinforcement of polymers. Their Nano-scale dimensions contribute enormous rheological benefits to the polymer resin systems. Nano-particles at very low concentration (<5 wt. %) dispersed in polymer resins often impart superior mechanical, thermal, barrier and electro-magnetic properties. So, nano reinforcement of polymer has opened up a new horizon for different property development and their potentials in structural applications.

According to the findings of Dubey, (2017), Sandra *et al* (2018), and Hassanzadeh-Aghdam *et al* (2018), silicate nanoparticles are produced and used in the polymer materials to create nanocomposites of polymer matrix (PMNCs) are among the current research areas of this field. These uniform dispersion of nanoparticles in the polymer matrix leads to a significant improvement of the properties of the nanocomposites as compared to those of conventional reinforced composites micro particles.

From these and other perspectives, polymer nanocomposites are relatively a new type of composite material, of which the possibilities are numerous, but they are not fully explored. Thermoplastic recycled PET reinforced with nano particle and producing dielectric materials are another relatively new development within the field of composites.

A dielectric material is a type of insulator which becomes polarized when it comes in contact with an electrical field. It can easily support an electrostatic field even though it is not a conductor of electricity. Such materials are used in many places such as in capacitors and radios, as well as transmission lines for radio frequency. It can be used to store energy too, if it is configured properly. Most of these materials are solid in nature, but some fluids and gasses also exhibit dielectric properties. An example of such a gas is dry air, while examples of solid dielectrics include mica, ceramic, plastics and glass. Distilled water is a dielectric liquid.

In this research, the results of the new combination of composites have been described. This combination consists of a recycled PET reinforced with nanocly in different loading. To successfully produce this new type of nanocomposite and to understand their mechanical and dielectric properties, the individual components need to be understood at first. For this reason, most of the researchers have been focused on the nanocomposite matrix material, followed by an investigation of the properties of nano polymer.

Hence, the goal of this research is to improve the properties of the recycled PET by reinforcement on the nanometer level and thereby upgrading the properties of this relatively low cost thermoplastic composite.

1.2 Statement of the Problem

Investigating and discovering of composite materials in different scales are among the prominent interests of scholars of this time. Because, according to Kurahatti *et al* (2010) findings, nanotechnology have a power to deliver and develop new properties and structures which will result in increased performance (e.g., Nano-energetics and new types of catalysts), reduced cost of maintenance (e.g., wear reduction, self-healing and self-repair), enhanced functionality (e.g. adaptive materials), new types of electronic/opto-electronic/magnetic material properties and increasing mechanical and physical properties.

Due to their versatile applications and widespread properties, polymer Nano-composites annual production increase as a result a large amount of composite material waste has also been generated. Hence, some materials are difficult to disposal and recycle, for example according to Yao *et al* (2019) due to the high modulus and corrosion resistance of carbon fiber composites has led to the difficulty of disposal and utilization of waste materials.

Due to the purpose of this research work, for producing insulation materials, the mechanical dielectric properties of the materials used is of concern because of the role it plays in the useful life of the structures under static and electric loading. It is obvious that in order to improve the reliability and quality of the insulating materials, it is at first essential to be aware of the possible raw materials, sample fabrication method, testing standards and testing methods that have been imparted to a samples when it is used for structural application.

Much of the valuable data have been published mostly on the mechanical and dielectric properties of recycled PET materials without Nano particle infused polymers. However, the comprehensive study on the mechanical and electric properties of recycled PET with nanoclay infused matrix has not reported. Further, it is reported that, among the several parameters that affect the structure of fabricated parts, tensile loading, flexural loading, dielectric strength and dielectric constant are among the most important ones that are worth for investigating in the applications. Therefore this work aims to perform a detailed experimental investigations on the mechanical and dielectric properties of recycled PET with nanoclay infused polyester matrix for evaluating the effect of the nanoclay loading on the mechanical and dielectric properties in terms of static, dielectric and microstructural analysis.

1.3 Objectives of the Study

1.3.1 General Objective

The main focus of this project is to synthesis and characterization of composite materials a Na^+ Montmorillonite clay incorporated Recycled Spring Water Bottle (PET), and to characterize mechanical properties of the developed material.

1.3.2 Specific objective

- To Manage the Municipal Solid Waste: Management of solid waste is an important problem in today's world. The amount of plastics consumed annually has been growing steadily.
- To Recycle the Spring Water Bottle: Polymer recycling is a process by which polymers are converted into useful products rather than landfills.
- To synthesis and characterize the multi walled carbon nanotube clay with incorporated Spring Water Bottle : This project aims at recycling of Polyethylene Terephthalate (PET), which is a major post-consumer contributor of Municipal Solid Waste (MSW), to produce a new dielectric material using nanotechnology in different loading levels of 0%, 2%, 4% and 6% with recycled PET matrix system
- To carry out experimental analysis (Tensile, Flexural, Impact, Hardness, Dielectric strength, Dielectric constant and Volume resistivity) of the fabricated composites samples.
- To conduct microstructural study on 0%, 2%, 4% and 6% nanoclay samples (For before and after fracture) through X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDS) and Scanning electron microscopy (SEM) analysis.

1.4 Scope of the Present Study

Great advances are taking place in the development and application of newer polymer composite materials, to enhance the:

- Higher design flexibility, which enables them to attain desired mechanical and physical properties by varying the composition of constituent materials.
- Properties such as high strength, high thermal resistance, low coefficient of moisture absorption, dielectric properties and excellent resistance to chemicals and corrosion.
- Wide range of possible applications, ranging from military aircraft, naval submarines, and aerospace structures to automobile components and sports equipment.

- High strength to weight ratio, which results in reduction of costs due to decrease in weight of structures used in the applications mentioned above.

To meet this demand, in recent years, a new Nano polymer matrix material has been developed on the basis of a new static and dynamic mechanism. For some polymer matrix composites, the static and dynamic properties are improved by adding the Nano particle to the polymer system. Nanoclay is usually used as Nano particle to form the nanocomposites in the polymer matrix. It is well known that the nanoclay reinforcement will improve the static and dynamic property of the composite greatly, if the reinforcement is chosen properly and combined with the matrix in a special way, to make varied mechanisms by playing roles together in the composite. Thus, the work throws light on the studies on the mechanical and electrical properties of the recycled PET with varying nanoclay loading, and analyzing the effect of nanoclay loading and on the properties.

Chapter II: Literature Survey

2.1 Introduction

Poly (ethylene terephthalate) (PET) is one of aromatic polyesters, widely used polyester resin in conventional industry because of its good mechanical properties, low cost, high transparency, high process ability, and moderate recyclability. Thus, PET holds a potential for industrial application, including industrial fibers, films, bottles, and engineering plastics (Jun and Seong, 2012). However, due environmental unfriendly and waste disposal management problems the global, currently, researchers, governmental and nongovernmental institutions focus is on creating and expansion of the green economy. Accordingly, among the prominent areas that, the disposal of any material seeing is to be based on the strategy of the four R's: reduction (combating waste generation), reuse (of materials), recycle (use of discarded material in new products) and recover (energy extraction from discarded waste) (Luiz *et al*, 2017).

The studies on the mechanical and electrical characterization such as flexural strength, flexural modulus, tensile strength, tensile modulus, impact strength, Dielectric Strength, Dielectric constant, volume resistivity properties etc., of PET polymer and nano PET polymer matrix composites have been carried out by many researchers.

The studies on these mechanical and electrical characteristics are important, as these polymer based composites are difficult to characterize the material due to their anisotropic and non-linearity. The studies of these materials are needed to achieve near-net shape of the polymer based composites, recycled PET polymer based composites being applied in the structural area, due to its unique static and dielectrical characteristics.

In order to understand and access the current status of the researches regarding to the synthesis and characterization of composite materials from a clay incorporated PET, an extensive literature survey relevant to the present investigation is given below:

2.2 Composite Materials

The perceptions and understanding of composites by scholars are different due to their specific concern and their exposure. However, Nano-composite material has over the years broadened significantly to encompass a large variety of systems such as one-dimensional, two-dimensional, three-dimensional and amorphous materials, made of distinctly dissimilar components and mixed at the nanometer scale as stated by Charles (2013). According to Azonano (2009) nanocomposites are “materials with a nanoscale structure that improve the macroscopic properties of products.” Hence, nanocomposites are clay, carbon, or polymer, or a combination of these materials with nanoparticle building blocks.

Composite materials, thus providing significant improvement in terms of mechanical and electrical erosion reduction, mechanical strength enhancement, electrical breakdown/endurance behavior, and space charge mitigation (Wan *et al*, 2014).

The dimension and size of the materials affecting the whole properties and applications of materials. For example according to Luo and Daniel (2003) findings, the transition from micro-particles to nanoparticles yields dramatic changes in physical properties. Because, chemical and physical interactions are governed by surfaces and surface properties.

In the case of particles and fibers, the surface area per unit volume is inversely proportional to the material’s diameter, thus, the smaller the diameter, the greater the surface area per unit volume. In general, nanomaterials provide reinforcing efficiency because of their high aspect ratios. In addition, according to Park *et al* (2001), properties of a nanocomposite are greatly influenced by the size scale of its component phases and the degree of mixing between the two phases. Depending on the nature of the components used (layered silicate or nanofiber, cation exchange capacity, and polymer matrix) and the method of preparation, significant differences in composite properties may be obtained.

Nanocomposite materials have emerged as suitable alternatives to overcome limitations of micro-composites and monolithics, while posing preparation challenges related to the control of elemental composition and stoichiometry in the nanocluster phase (Pedro *et al*, 2009). Besides, According to Charles (2013), Nanocomposites can dramatically improve properties like: · Mechanical properties including strength, modulus and dimensional stability; Electrical conductivity; Decreased gas, water and hydrocarbon permeability; · Flame retardancy; Thermal stability and Chemical resistance.

The paradigm shift of producing materials from macro to Nano scale comes with the following substantial improvement properties such as: Mechanical properties e.g. strength, modulus and dimensional stability; Decreased permeability to gases, water and hydrocarbons; Thermal stability and heat distortion temperature; Flame retardancy and reduced smoke emissions; Chemical resistance; Surface appearance; Electrical conductivity and Optical clarity in comparison to conventionally filled polymers as stated by Pedro *et al* (2009). Further, other benefits from nanocomposites include improvement in modulus, flexural strength, heat distortion temperature, barrier properties, and other benefits and, unlike typical mineral reinforced systems, they are without the conventional trade-offs in impact and clarity.

In addition ss stated by Jun and Seong (2012), from both an economic and industrial perspective, the major challenges for high performance polymer nanocomposites are to fabricate the polymer nanocomposites with low costs and to facilitate large scale up for commercial applications.

Specifically to this research, according to Pedro and his group members, in plastics the advantages of nanocomposites over conventional ones don't stop at strength. The high heat resistance and low flammability of some nanocomposites also make them good choices to use as insulators and wire coverings. Another important property of nanocomposites is that they are less porous than regular plastics, making them ideal to use in the packaging of foods and drinks, vacuum packs, and to protect medical instruments, film, and other products from outside contamination.

Due to specific and general applications and contributions classification of the nanostructured materials are varied. They are classified due to their dimensionality, morphology, composition, and uniformity/ agglomeration states as explained by Liu *et al* (2011); besides, according to Saiful *et al* (2013) they classifieds *b*ased on dimensionality, nanostructured materials are classified into 1D (i.e., thin films or surface coatings), 2D (i.e., fixed long nanostructures, thick membranes with nanopores, and free long aspect ratio nanowires), and 3D (i.e., fixed small nanostructures, membranes with nanopores, and free small aspect ratio nanoparticles).

2.3 Polymer Nano composites

This types of nanocomposite materials have a widespread application due to its comprehensive properties. The unique combination of the nanomaterial's characteristics, such as size, mechanical properties, and low concentrations necessary to effect change in a polymer matrix, coupled with the advanced characterization and simulation techniques now available, have generated much interest in the field of nanocomposites (Pedro *et al*, 2009).

To distinguish and compare Nano and micro polymer materials scholars are using different criteria's. For example content of fillers, size of fillers, and specific surface area of fillers are used as stated by Tanaka *et al* (2004) and Wan *et al*, (2014).

According to Wan *et al*, (2014) explanation, polymer nanocomposites are defined as composites in which small amounts of nanometer sized fillers are homogeneously distributed in a polymer by certain weight percentages (wt%). Besides, according to findings of Sambarkar (2012) and Chapman and Mulvaney (2001), the notion of using nanoparticles as fillers in polymer materials have conveyed the attention of researchers due to their exclusive mechanical, electrical, optical, and thermal properties.

In addition, according to Yao *et al* (2019) findings, conductive polymer composites have a range of excellent properties, like high conductivity, high specific strength, high specific modulus, high temperature, corrosion resistance, fatigue resistance and so on. Hence, they can be used not only as a structural material to carry loads, but also as functional materials.

To improve the basic properties and enhancing its application researchers are coming up with the incorporation of Nano fillers like Nano clays, Nano particles, nano tubes, nano fibers, etc., to polymer nanocomposite materials, additionally, this incorporation of nano reinforcements into elastomers, which considerably enhances their mechanical and thermal barrier properties in conjunction with noticeable improvements in adhesion, rheological and processing behavior. Furthermore, better dispersion of these fillers within the matrix provides high performance Nano composites and also the properties of the Nano scale filler are significantly higher than those of the base matrix (Pinnavaia and Beall, 2000; Anandhan and Bandyopadhyay, 2011; and Senthil, 2014).

Materials properties are influencing by different factors: among the common as stated by Farzana *et al* (2006) are increased relative surface area and quantum effects; and according to Christopher and Lerner (2001) nanocomposites properties predominated by the interfacial interactions and quantum effects associated with Nano dimensional structures.

Besides, according to the findings of May-Pat *et al* (2011), Nanocomposite materials often possess a combination of physical properties that are not present in conventional polymer matrix composites. Because of their high aspect ratios, adding low concentrations of carbon Nano - tubes (CNTs) into a polymer matrix can improve the mechanical, thermomechanical and electrical properties of these polymer composite materials.

In addition, two key issues necessary to achieve superior performance in CNT filled polymer composites are a homogeneous distribution and dispersion of the CNTs inside the polymer matrix and a strong interaction between the CNTs and matrix. One of the most common methods used to disperse CNTs in a thermoplastic polymer matrix is melt blending.

Furthermore, more related with the current research work, as elucidated by Kurahatti *et al* (2010), by using fillers reinforcement of polymers whether inorganic or organic, is common in the production of modern plastics. Polymeric nanocomposites (PNCs) represent a radical alternative to the conventional filled polymers or polymer blends. In contrast to conventional systems, where the reinforcement is of the order of microns, PNCs are exemplified by discrete constituents of the order of a few nanometers (<100 nm) in at least one dimension. The small size of the fillers leads to an exceptionally large interfacial area in the composites. The interface controls the degree of interaction between the filler and the polymer and thus controls the properties.

There are different classification of polymer nanocomposites among the common and broadly according to Kurahatti *et al* (2010), Nanoclay-reinforced composites, Carbon nanotube-reinforced composites, Nanofiber-reinforced composites, and Inorganic particle-reinforced composites.

There are different manufacturing of methods and process of Polymers. For example according to Ogata *et al* (1997) and Saiful *et al* (2013) Polymer Matrix Nanocomposites (PMNC): are preparing by layered materials and those containing CNTs. As elucidated by Azioune *et al* (1999), Ogasawara et al (2004) and Saiful *et al* (2013), the most important ones are i) Intercalation of the polymer or pre-polymer from solution; ii) In-situ intercalative polymerization; iii) Melt intercalation (Figure 1); iv) Direct mixture of polymer and particulates; v) Template synthesis; vi) In-situ polymerization; and vii) Sol-gel process. Publications dealing with various methods for the incorporation of nanodispersoids into conducting polymers are also available.

According to findings of Pukanski and Fekete (199), production process are among the critical factors that play a main role in the performances and properties of nanocomposites are particle-particle interactions and particle-matrix interactions. The particle-particle interaction deteriorates the composite's performances, whereas the filler-matrix interaction produces a composite with improved performances. The attraction and repulsion between the particles, due to van der Waals and electrostatic forces, affect the particle-particle interaction in a composite as stated by Russsel *et al* (1989). In addition according to Zhang *et al* (2003) result shows that, the reduction in the repulsive forces between the particles can be achieved by decreasing the particle size in the nanoscale. The adverse impact of the particle-particle interaction can attribute to particle aggregation and, consequentially, the performance of the nanocomposite may deteriorate.

Besides of the above as explained by Saiful *et al* (2013) chemical modification methods like in situ processing method, allow controlling "morphology" and "polymer-nanofiller interface" in polymer nanocomposites. Hence, the interfacial area between the nanofillers and the polymer controls the properties of a polymer nanocomposite. The chemical properties of this region affect the polymer chain mobility, degree of curing, and the crystallinity.

Furthermore, manufacturing of Nano-phased structural polymer composite materials can used differently, among the common conventional fabrication process as elaborating by Mazumder (2002) and Farzana *et al* (2006) are wet lay-up, pultrusion, resin transfer molding (RTM), vacuum assisted resin transfer molding (VARTM), autoclave processing, resin film infusion (RFI), prepreg method, filament winding, fiber placement technology, etc.

Controversially, with the application of composite materials and the increase of annual production, a large amount of composite material waste has also been generated. In particular, the high modulus and corrosion resistance of carbon fiber composites has led to the difficulty of disposal and utilization of waste materials (Yao *et al*, 2019).

2.4 Dielectric properties of polymer composites

The definition and explanation of dielectric materials according to the focus of scholars is different. However, the common is: dielectric material) is an electrical insulator that can be polarized by an applied electric field. When a dielectric is placed in an electric field, electric charges do not flow through the material as they do in an electrical conductor but only slightly shift from their average equilibrium positions causing dielectric polarization. Because of dielectric polarization, positive charges are displaced in the direction of the field and negative charges shift in the direction opposite to the field (for example if the field is moving in the positive x-axis, the negative charges will shift in the negative x-axis). This creates an internal electric field that reduces the overall field within the dielectric itself. If a dielectric is composed of weakly bonded molecules, those molecules not only become polarized, but also reorient so that their symmetry axes align to the field. The study of dielectric properties concerns storage and dissipation of electric and magnetic energy in materials (Thoms *et al*, 2017; Muhammad *et al*, 2005).

Specifically, polymer composites, according to Hristiyan *et al* (2009) perceptions, due to their unique mechanical properties such as flexibility and light weight, combined with versatile chemistry (composition), process ability, and simple manufacturing polymers exploited as candidate dielectric Materials. Furthermore, according to the findings of Muhammad *et al* (2005), polymer composites in the solid or viscoelastic state, the physical structure is a great importance for determining the dielectric behavior.

Jinkai *et al* (2016) the classification of Materials can be done through dielectric constant values, because the dielectric constant *k*, is a physical feature related to the electric polarizability of a material. The dielectric constant k is the relative permittivity of a dielectric material. It is an important parameter in characterizing capacitors. It is unfortunate that the same symbol k is often used for Coulomb's constant, so one must be careful of this possible confusion. It is more typical of physics texts to use the form $1/4\pi\varepsilon_0$ for Coulomb's constant. Physically the greater the polarization developed in the materials, the higher the *k*. In comparison to the *k* value of silicon dioxide as an example, which is 3.9, the dielectric materials are classified into high-*k* dielectrics ($k > 3.9$) and low-*k* dielectrics ($k < 3.9$). Whereas Polyethylene is the most common plastic (primary use is in packaging (plastic bags, plastic films, geomembranes, containers including bottles, etc.) dielectric constant is 2.25.

According to the findings of Yao *et al* (2012), Wu *et al* (2002) and Li *et al* (2007) shows, traditional dielectric materials are ceramics (e.g., mica, silicon dioxide, and BaTiO3) played a role in producing of elcetronic devices, but due to their poor flexibility, high-density, and challenging processing conditions. Considering these limitations, polymeric dielectrics have been broadly explored as alternative dielectrics recently.

For instance, high *k* dielectrics can serve as effective materials for cable insulation (Huang and Jiang, 2015), charge-storage capacitors (Dang *et al*, 2013), electro caloric cooling (Zhang *et al*, 2015), and artificial muscles (Brochu and Pei, 2010). Moreover according to the findings of Maier (2001), low-*k* materials are the key insulating components for isolating signal-carrying conductors from one another as well as reducing the signal propagation delay and dynamic power dissipation in the high-density and high-speed microelectronic packaging.

As stated by Jinkai *et al* (2016), polymer nanocomposites consisting of nanoparticles allow for a large tenability of the dielectric constant by varying the identity, shape, and content of Nano additives, as well as engineering the nanoparticle–polymer interfaces. However, challenges still exist today on rationally designing optimum composite microstructures to realize a target high-*k* or low-*k* value.

2.5 Nano particle reinforced thermoplastic composites

As elucidated by Adel (2012), plastics are the basic ingredients of animal and plant life and form a part of the larger family called polymers. They offer advantages such as low density, lightness, transparency, resistance to corrosion and color, their applications vary from domestic articles to medical instruments. Plastic are broadly classified into; (i) thermoplastics and (ii) thermosetting plastics. Both are long chain-like molecules but differs in their bonding. In thermoplastics the long chain molecules are held together by weak vander waal forces where as in thermosetting, the long chain molecules are held together by strong bonds. Thermoplastic can be molded into any shape. Thermoplastics differ from thermosetting polymers. Thermosets form irreversible chemical bonds during the curing process.

Thermoplastic polymers are come up monomers together via electrical bonds called van der Waals forces that weakly attract neutral molecules to each other. These repeating units arrange themselves in such a way that thermoplastic polymer molecules look like many strands of pearls mixed together. Thermoplastic materials have many features. Some products made from thermoplastic materials are used for electronic applications. They protect against electrostatic discharge and radio frequency interference. Some of the most common types of thermoplastic are polypropylene, polyethylene, polyvinylchloride, polystyrene, polyethylenetheraphthalate and polycarbonate

Polyethylene is a thermoplastic polymer consisting of a long hydrocarbon chain. Most polyethylenes, such as low density polyethylene (LDPE), linear low density polyethylene (LLDPE), crosslinked polyethylene (XLPE), and high density polyethylene (HDPE), have a great resistance to electrical stress, thus making them useful as high voltage insulating material besides their primary use as packaging material, such as plastic (Wan *et al*, 2014).

Many researchers have used MMT and CNT nanoparticle as filler in thermoplastic such as Polypropylene (PP), Polyethylene Terephthalate (PET), Polyvinyl Chloride (PVC) etc.

composites. Some of the important research work on nanoparticle based thermoplastic polymer composites are summarized below.

Accordingly, according to Tanaka *et al (2006),* Arief et al (2013) and Makmud *et al* (2012) polyethylene are used in investigating high voltage insulation as well as in applications. Moreover, fiber-reinforced thermoplastics over metals and ceramics, that have been recognized for years, are improved fracture toughness, impact resistance, strength to weight ratio, as well as high resistance to corrosion and enhanced thermal and fatigue properties that have often been put in good use for practical applications in the aeronautic, automotive, and energy sectors (Marsh, 2003). Additional nanoscale fillers, such as carbon nanotubes (CNTs) have been mixed with CFs to reinforce polymer matrices. Their high specific surface area enables the formation of a large interphase in the composite and strong filler-matrix interactions. In the same way, the addition of nanoclays to fiber-reinforced thermoplastic composites has been reported to improve damping properties, fatigue life, toughness, and wear resistance as explaining in detail by Mohammed and Ana (2014), Qian *et al* (2010) and Lin et al (2012).

Hence according to the findings of Mohammed and Ana (2014), the synergetic effect of CFs with the inorganic nanoparticles is believed to be the major cause for the mechanical improvement achieved.

Besides, Lee *et al*. (2010) have developed clay particles reinforced wood fiber/plastic composites (WPCs) to improve their mechanical and flame retardancy properties. A high degree of exfoliation for nanoclay in the wood fiber/high density polyethylene (HDPE) composites was successfully achieved with the aid of maleated HDPE (PE-g-MAn), through a melt blending master batch process. They used X-ray diffraction (XRD) and transmission electron microscopy to determine the structures and morphologies of the composites respectively. Also the effects of clay content and degree of clay dispersion on the mechanical and physical properties and flame retardancy of wood fiber/HDPE composites that contained a small amount of clay, in the range of 3–5 wt % are presented. They have concluded that achieving a higher degree of dispersion for the nanosized clay particles are critical to enhance the mechanical properties and the flame retardancy of WPCs when small amounts of clay are used.

Najafi *et al* (2012) have investigated the effect of nanoclay on the physical and mechanical properties of nanocomposites based on polypropylene, reed fiber and maleated polypropylene (MAPP) as coupling agent. The mass ratio of reed fiber to polypropylene was controlled at

60/40 for all blends. The concentration was varied and set to 0, 2 and 4 per hundred compounds (phc) for nanoclay and 0 and 2 phc for MAPP. Results indicated that the tensile modulus and strength of PP/reed flour composites significantly increased with nanoclay loading. However, the impact strength and water uptake of the composites decreased by 4% addition of nanoclay. Additionally, they found that the coupling agent is improved the mechanical and physical properties of the composites by morphological study.

Hybrid composites of polypropylene (*PP*)/nanoclay (*NC*)/glass fiber (*GF*) were prepared by Rahman *et al* (2011) through extrusion and injection molding process. They found that, the incorporation of nanoclay into improves the thermal stability, flexural strength, flexural modulus, tensile strength of the hybrid material fabricated. Further, Nafchi *et al* (2012) have investigated composites based on Wood fiber/nanoclay/polypropylene and found that Nanoclay addition improves both the mechanical and physical properties. It also lowers the water absorption properties with relative to pure composite.

Manohar and Yoush (2015) have studied both mechanical (flexural and tensile strengths) and physical properties composites based on Wood flour/nanoclay/ polypropylene and found improved properties by the incorporation of nanoclay. The water absorption properties significantly reduced in nanoclays filled composite.

2.6 Nano particle reinforced recycled thermoplastic composites

Currently, the growing consumption of polymer products creates the large quantities of waste materials resulting in public concern in the environment and people life. Recently, many researchers have tried to develop a new science for polymer recycling using nanotechnology. Because, material recycling is a term used to describe processes in which the macromolecular structure is kept basically intact, and the material is reformed into a new product. In this section, the application of different nanoparticle in the recycled polymers (PET, PP, HDPE, PVC, etc.) with attributed composites and blends are studied.

Polymer recycling would be any process which diverts the polymer waste to any place or use, other than landfill, but its options could be far from optimal as a result of economic (e.g. very expensive) and environmental (e.g. high energy consumption) reasons. According to Burillo *et al* (2002) findings shown that, chemical recycling refers to the decomposition of the macromolecular structure to generate low molecular weight compounds. Incineration is a third category of polymer waste utilization where the product is heat used in the production of electricity. As stated by Väisänen *et al* (2016), Boota *et al* (2015) and Yao *et al* (2019), there are two main sources of polymer composite waste: one is the waste in the process of production and molding, such as prepreg materials, expired materials, scrap materials, unsuitable parts, flash edges, test waste, etc.

In addition, according to Hence, Metin (2003), recycling of plastics is an important industrial application because it is critical in influencing the environment, so, purpose of recycling is to decrease the amount of waste by recirculation of raw materials and to improve the material utilization. Hence, due to the findings of the above scholar, there are four main types of methods used specifically for plastic recycling, namely primary, secondary, tertiary and quaternary. In the primary recycling, there is a conversion of scrap plastics by one or a combination of products having performance characteristics equivalent to the original products made of virgin plastics. In the secondary one, the conversion occurs with yielding products having less demanding performance requirements than the original material. Melt recycling is considered as a secondary recycling. There is a process involving chemical treatment in tertiary recycling producing chemicals and fuels from scrap or waste plastics. The quaternary recycling involves process technologies of recovering energy from scrap or waste plastics by incineration.

Furthermore, besides of the above according to different scholars the following literatures presented current status of nanotechnology in the polymer recycling and their related composite blends. Basilia *et al (2011)* have synthesized Recycled polyethylene terephthalate (RPET)/organo-montmorillonite nanocomposites by direct melt intercalation method. They reported that the thermal degradation has been decreased and tensile strength increased with the increase in clay loading. 5 wt.% of synthesized Philippine MMT (Na-MMT) offered the best improvement of tensile properties. The mechanical behavior of the nanocomposites at various clay loadings (Fig. 30) shows that the higher the clay content the better is the tensile strength of the samples. Furthermore, Giraldi *et al* (2008) have found that the optimized values of strength and toughness were obtained at 2.5 wt. % of nanoclay instead of 5 wt. %.

Pegoretti *et al* (2004) have prepared the rPET/organo clay nanocomposites at several nanoclay content (1, 3 and 5 wt.%). This works suggested the highest stiffness and tensile strength at 5 wt.% of nanoclay. Basilia *et al* (2011) have also studied same composition obtained same result while, Tg slightly decreased by the clay content, may be due to clay agglomeration which occurred above a critical clay loading.

Youngjae *et al* (2004) have prepared the recycled PVC/clay nanocomposite by melt mixing of recycled PVCs and modified clays. In the case of 10 wt.% of nanoclay, the characteristic peak of clay was purely disappeared. Also, the coefficient of thermal expansion (CTE) decreased with addition of nanoclay with rPVC. The improvement of mechanical properties of the nanocomposite is also observed simultaneously at, 1, 3, 5 and 10 wt.% of nanoclay loading Further, the thermal decomposition behavior and linear dimensional of nanocomposite improved proportionally to the contents of nanoclay. Further, it is observed that, at 55 ºC, the storage modulus of nanocomposite composed of 10 wt.% of nanoclay was 11 times higher than that of neat rPVC. Fig. 31 shows the storage modulus of the nanocomposites as a function of temperature.

Jannapu (2010) have investigated the effect of graphene nanoparticles on the properties of recycled High Density Polyethylene (rHDPE). The large improvement of properties was observed only with a little content of graphene reinforcement. It was reported that only about 0.25 wt.% of graphene introduced more tensile modulus, dielectric constant (k) and thermal conductivity were obtained in rHDPE-nanocomposite. It is concluded that only extremely slight contents of nanofillers can significantly improve the properties of rHDPE.

Suresh *et al.* (2017) have investigated the nanocomposite of Recycled polymethyl methacrylate (rPMMA) with both natural and organically modified silicate layers. There was no improvement in thermal properties of nanocomposites but at higher dispersion degree, the nanocomposites showed higher transparency extent. The tensile modulus increased with nanoclay content for both nanoclays which introduced nanocomposites with attractive properties by the maintenance of transparency and thermal properties of waste matrix.

Andricˇic´ *et al* (2008) prepared of composites consisting of preconsumer waste composed of PVC and PP in the presence of surface-modified nanosized $CaCO_3$ contents of 40, 25, and 10 wt. %, respectively. Agglomeration of the nanofiller in matrix is studied by SEM. The compatibility between new interlayer has been developed by nanofiller. They found that the tensile strength of PVC sheets containing 10, 20 and 30 wt. % of waste laminate (1, 2 and 3 wt.% of PP) decreased as PP content increased. In the samples with lower PP content, mechanical properties got better as the filler content increased, while with higher PP content, mechanical properties reached its maxima at about 6 wt.% of CaCO3 content.

2.7 Strategy on Characterization of Recycled Pet Nano Composites

As already been stated elsewhere in this thesis, for dielectric materials, the mechanical and electrical properties of the materials used is of concern because of the role it plays in the useful life of the structures under static and dynamic loading. It is obvious that in order to improve the reliability and quality of the insulating materials, it is at first essential to be aware of the possible raw materials, fabrication method, testing standards and testing methods that have been imparted to a samples when it is used for structural application. Hence, it is also necessary to study the impacted structures to ensure the stiffness required for withstanding the static and dynamic loading.

Much of the valuable data have been published mostly on the mechanical and electrical properties of virgin PET without nano particle infused polymers. However, the comprehensive study on the mechanical and electrical properties of recycled PET with nanoclay infused matrix has not reported.

Therefore this work aims on the following:

- To perform a detailed experimental investigations on the mechanical and electrical properties of recycled PET with nanoclay for evaluating the effect of the nanoclay loading on the mechanical and electrical properties in terms of static, dynamic and microstructural analysis.

2.8 Need for the Present Study

The polymer based composite materials have been used in building dielectrical materials but they continually suffer from static loading, dynamic loading and environmental effect, and are in service for many decades now. It is also critical that, the most stringent quality measurements are needed to study the mechanical properties of fabricated composites. Reliability is an important criterion in the manufacture of dielectrical materials, and therefore, these composite manufacturers need to maintain high-quality on a consistent basis.

Hence, this research work focuses on the manufacturing of high performance composites which are discussed above and evaluation of mechanical and electrical properties such as tensile, flexural, impact, hardness, dielectric strength, dielecrtic constant and volume resistivity using experimentally. The microstructures of the fabricated laminates before and after fracture are assessed by using XRD, EDS and SEM analysis.

Chapter 3: Materials and Methods

3.1 Introduction

There are several factors which have been considered while selecting the materials for a composite structure. The most important factors are clay and PET chemistry, dispersion of the clay in PET, compatibility of clay and PET matrix, matrix formulation, processing parameters and curing conditions. In this chapter, the selected materials, their physical properties, dispersion method of nanoclay in matrix, sample manufacturing method and experimental methods for mechanical and electrical characterizations are described.

3.2 Materials Selection

In this thesis, the following materials have been used for the manufacturing of composite samples.

Matrix Material: Recycled poly (ethylene terephthalate) (rPET) pellets (intrinsic viscosity ASTM D 4603-91 5 0.69 dL/g) produced by plastic recyclying machine (shown Fig 1) by extruding reclaimed beverage bottles were utilized. To minimize the polyester hydrolysis by moisture presence, the recycled PET pellets were dried at 1308C for 6 h before the extrusion.

Figure 1: Recycling of PET bottles

Clay: The commercial nanoclay used in this study is provided by Southern Clay Products, Na$^+$ Montmorillonite (unmodified having CEC 92.6meq/100 g clay) modified with some concentration and octadecyltrimethyl ammonium chloride. The specific gravity and mean particle size of Cloisite Na$^+$ are reported as 2.86 and 6 μm, respectively, by the supplier. According to the x-ray diffraction results provided by the supplier, the gallery spacing of Cloisite Na$^+$ is 11.7A°and having CEC 2.6meq/100 g clay. Figure 2 shows the commercial nanoclay, which has been used for the present study. The physical properties of the nanoclay used in this study are given table 1.

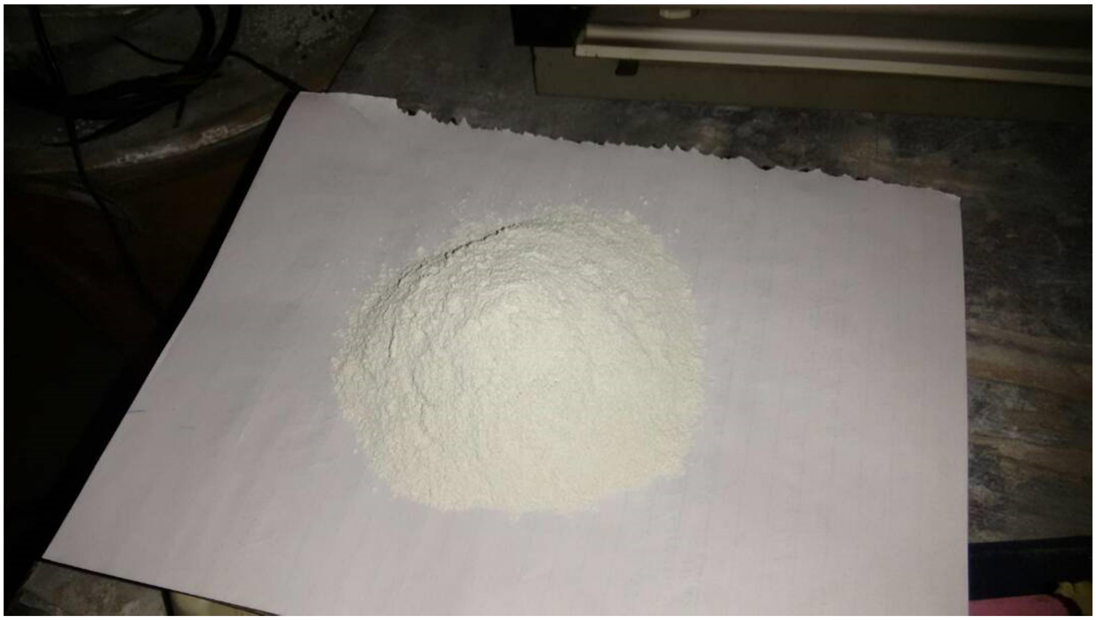


Figure 2: Photograph view of Nanoclay

Table 1: Physical Properties of Cloisite® Na$^+$ (Nanoclay)

Physical Properties	Metric
Specific Gravity	2.86 g/cc
Bulk Density	0.1994 g/cc
Loss On Ignition	7.00 %
Particle Size	<= 2.00 μm

3.2.1 Preparation of Organic Montmorillonite (OMMT)

Na$^+$ -MMT is dispersed in distilled water with some concentration and octadecyltrimethyl ammonium chloride is vigorously stirred for few times at a given temperature. The white precipitates are washed with hot distilled water (above 80 ^{0}C) until no bromide ion has been detected with a 0-1 mol/l $AgNO_3$ solution. The product obtained is then vacuum- dried at 70 ^{0}C to a constant weight and then it has been grounded and screened with 300-mesh sieve to get the modified clay (OMMT).

3.3 Synthesis and Preparation of Nano Composites

The composites prepared for the present study are 0% clay recycled PET composite sample and 4% nanoclay reinforced PET composite samples. Figure 3 and 4 show the machine that have been used and photographic view of preparation recycled PET composite pellets using twin screw extruder respectively.

Figure 3: Photograph view of machine used for recycling PET

Figure 4: Preparation of recycled nano composites pellets

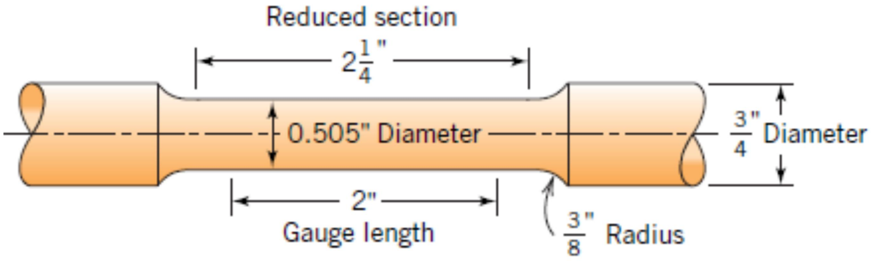
The sample dimensions and loading rates for all types of samples are given in table 2. Five replicate specimens are used for each test and the data reported are the average of five tests. Corresponding standard deviations along with measurement uncertainty values for the experimental data showing the maximum standard deviation are also included.

Table 2: Dimensions of the samples for various tests

Type of test	Width (mm)	Thickness (mm)	Length (mm)	Diameter (mm)	Span length (mm)	Crosshead speed (mm/min)	ASTM Standard
Tensile test	**25**	**3**	**200**	**-**	**150**	**1.5**	**A 638STM D**
Flexural test	10	3	150	**-**	**64**	**2**	**ASTM D790**
Charpy Impact test	12.5	8	120	**-**	**-**		**ASTM D256**
Hardness test	25	8	25	**-**	**-**	**-**	ASTM: D2240
Dielectric strength		**3**		**60**			**ASTM D149**
Dielectric constant		**3**		**110**			**ASTM D150**
Volume resistivity		**3**		**110**			**ASTM D257**

Figure 5 (a-d) Specimens of different samples

All samples are prepared with nanocly reinforcement of 0%, 2%, 4%, 6% with recycled PET materials. For measuring the tensile test and flexural test samples researchers prepared a sample based on ASTM standard respectively as presented on the following figure (William and David, 2014)



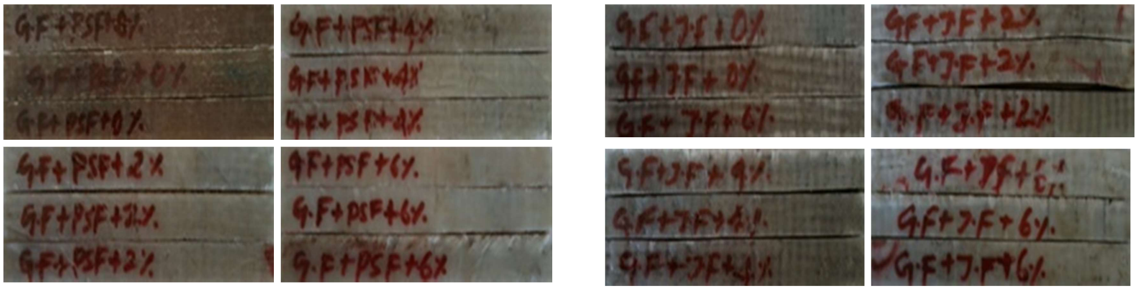


Figure 5: (a-b) Tensile and flexural test specimen samples

3.4 Synthesis and Fabrication of PMCs

According to the findings of Ji (2013), majority of the world's PET production is for synthetic fibers (in excess of 60%), with bottle production accounting for about 30% of global demand. In the context of textile applications, PET is referred to by its common name, polyester, whereas the acronym *PET* is generally used in relation to packaging. So, in this section, the advanced composite fabrication processes are discussed in more detail. Since the advanced composite industry is relatively new and still developing, other processes have been developing or changing to meet new performance requirements. Hence, some of the fabrication process used in the advanced composite industry is discussed in this section.

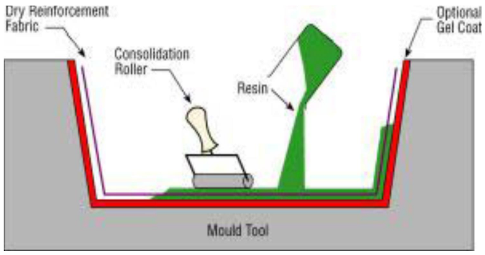
Hand lay-up is a simple method used for composite production. A mold is used for hand lay-up parts, where the composite is joined directly to another structure. The mold is as simple as a flat sheet or it has infinite curves and edges. For some shapes, molds are joined in sections so that they take apart for part removal after curing. Before lay-up, the mold is prepared with a release agent to insure that the part is not adhere to the mold. Reinforcement fibers have been cut and laid in the mold. It is up to the designer to organize the type, amount and direction of the fibers, which is being used. Resin has been catalyzed and added to the fibers. A brush, roller or squeegee is used to impregnate the fibers with the resin. The lay-up technician is responsible for controlling the amount of resin and the quality of saturation. Figure 6 shows the basic process of hand lay-up.

Vacuum bagging follows similar steps to the hand lay-up process. When the resin is adequately impregnated into the fibers with excess, the part is sealed with vacuum bagging materials. Figure 7shows the basic process of vacuum bagging. Vacuum resin transfer molding follows the first step of hand lay-up. The mold is prepared with a release agent, the fibers are cut and placed into the mold, and then new steps are taken. Figure 8 shows the basic process of resin transfer molding.

Compression molding is similar in theory but opposite in application to vacuum techniques. Figure 9shows several different designs of rubber bladders for compression molding. Filament winding is a process for composite production that usually involves a high initial investment. A machine has been purchased or created to wind filaments in an exact location on a mandrel.

Some advantages of filament winding are low material and labor costs and accuracy of product dimensions. Figure 10 shows the basic process of filament winding. Filament wound parts can be wound with or without resin.

Pultrusion is a composite fabrication process designed for structural shapes. The investment cost is very high and therefore only feasible for mass production parts. Fibers are drawn through a resin bath and then through a forming block. Figure 11 shows the basic pultrusion process. Many different factors are considered when forming a method of production. Table 3 gives a comparison of hand lay-up, filament winding and pultrusion.



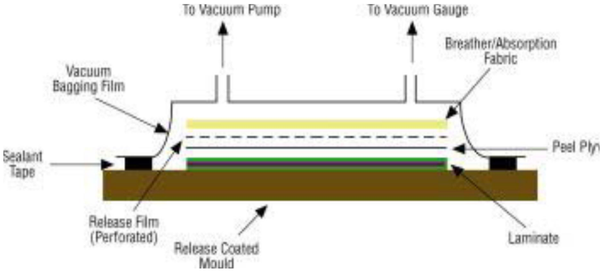
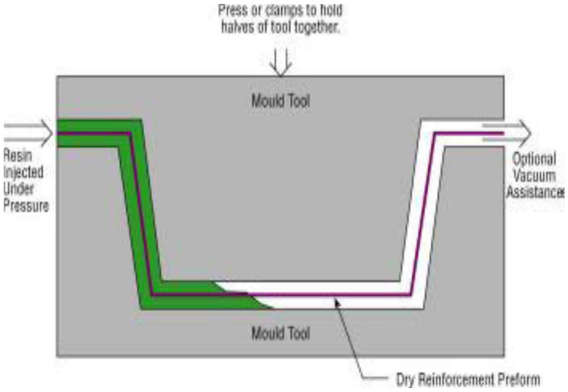


Figure 6:Basic Process of Hand lay-up

Figure 7: Process of Vacuum bagging



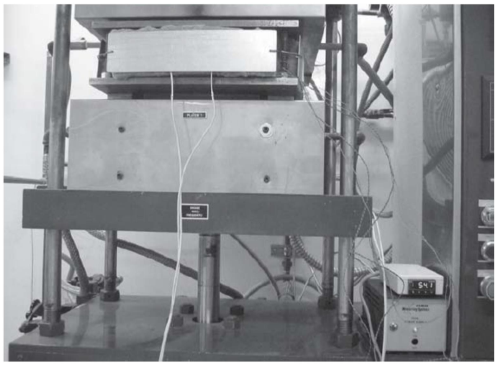
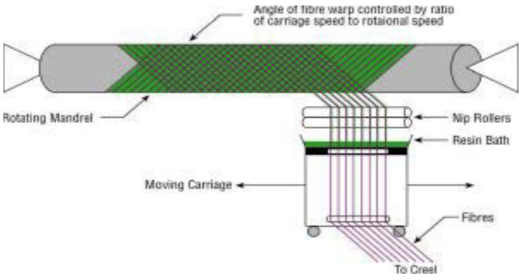


Figure 8: Vacuum resin transfer molding process

Figure 9: Compression molding process



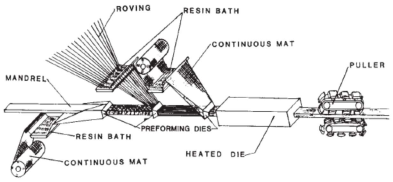


Figure 10: Filament winding process Figure 11: Pultrusion process

The above figures obtained from Vadivelvivek (2013) and https://netcomposites.com/guide/manufacturing/filament-winding/

Table 3: Comparison of fabrication process

Descriptions	Process type		
	Hand lay up	**Filament winding**	**Pultrusion**
Investment for single part	Low	High	Very High
Investment for multiple parts	Low	Medium	Medium
Investment for mass production	Medium	Low	Low
Labor cost	High	Medium	Low
Fiber resin ratio(no vacuum)	Poor	Good	Excellent
Fiber resin ratio(vacuum) Excellent	Excellent	Excellent	N/A
Complexity of part	Infinite	Mainly round	Cross section
Part tolerance	Good	Good	Excellent
Material usage	Poor	Good	Excellent
Speed of production	Slow	Medium	Fast
Part quality	Good	Excellent	Excellent
Strength orientation	Excellent	Excellent	Good

3.5 Materials Characterization

The development, modification, adoption, innovation and invention of materials that make our existence so comfortable has been intimately associated with the accessibility of materials. However, selection of a specific material for a particular purpose is a very complex process and it expects to have deep understanding in the basic properties, application, production process and quality of materials.

Therefore, to determine the basic properties, production process and quality of materials characterizing using different techniques is mandatory. In general there two ways of approaching materials characterization. The first of these is in terms of the engineering properties of materials, and reflects the need to know the physical, chemical and mechanical properties of the material before we can design an engineering system or manufacture its components. The second form of characterization is the microstructural characterization of the material (David and Wayne, 2008; Yang, 2008).

3.5.1 Mechanical Characterization of Fiber PMCs

The performance of fiber-reinforced polymer composites, with an emphasis on the general trends has been observed in their properties and behavior. Material properties are usually determined by conducting mechanical and physical tests under controlled laboratory conditions. The orthotropic nature of fiber-reinforced composites has led to the development of standard test methods that are often different from those used for traditional isotropic materials. These unique test methods and their limitations are discussed in relation to many of the properties considered in this chapter such as tensile, flexural, impact and dynamic properties. The effects of environmental conditions, such as humidity and hardness on the physical properties of composite laminates have been presented at the end of the chapter.

Tensile testing utilizes the classical coupon test geometry as shown (Figure 12) below and consists of two regions: a central region called the gauge length, within which a failure is expected to occur, and the two end regions which are clamped into a grip mechanism connected to a test machine. Flexural properties, such as flexural strength and modulus, are determined by bending test. In this test, a composite beam specimen of rectangular cross section is loaded in either a three-point bending mode (Figure 13(a)) or a four-point bending mode (Figure 13(b)).

The impact properties of a material represent its capacity to absorb and dissipate energies under impact or shock loading. Charpy and Izod impact tests are performed on commercially available machines in which a pendulum hammer is released from a standard height to contact a beam specimen (either notched or unnotched) with a specified kinetic energy. A horizontal simply supported beam specimen is used in the Charpy test (Figure 14a), whereas a vertical cantilever beam specimen is used in the Izod test (Figure 14b). The drop -weight impact test uses the free fall of a known weight to supply the energy to break a beam or a plate specimen (Figure 15). Energy absorbed by the specimen is calculated as

$$U_t = W/2g\ (u_1^2 - u_2^2)$$

where

W = weight of the striking head
u_1 = velocity of the striking head just before impact[= $(2gH)^{1/2}$]
u_2= measured velocity of the striking head just after impact
H = drop height

The compression- after-impact test is used for assessing the non-visible or barely visible impact damage in composite laminates. In this test method, an edge-supported laminated plate is impacted at the center with various energy level. After nondestructively examining the extent of impact damage (e.g. ., by ultrasonic C-scan), the plate is compression- tested in a fixture with antibuckling guides (Figure 16).

The damping property of a material represents its capacity to reduce the transmission of vibration caused by mechanical disturbances to a structure. A high value of damping factor is desirable for reducing the resonance amplitude of vibration in a structure. Fiber-reinforced composites, in general, have a higher damping factor than metals. However, its value depends on a number of factors, including fiber and resin types, fiber orientation angle, and stacking sequence.

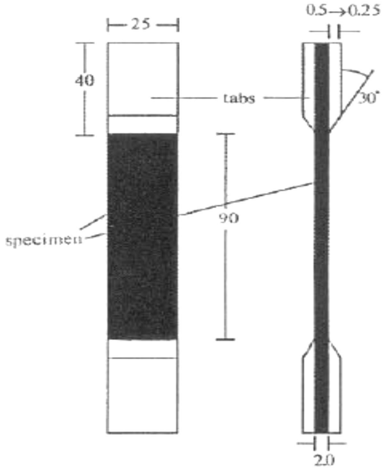
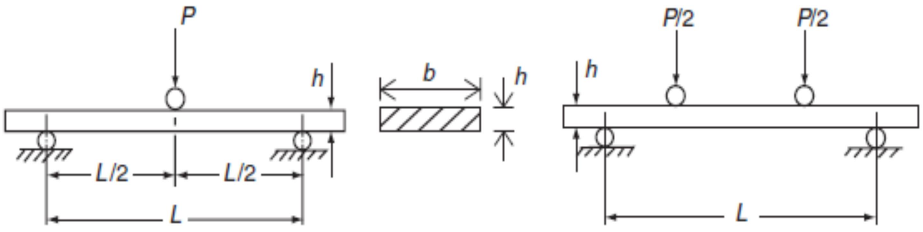
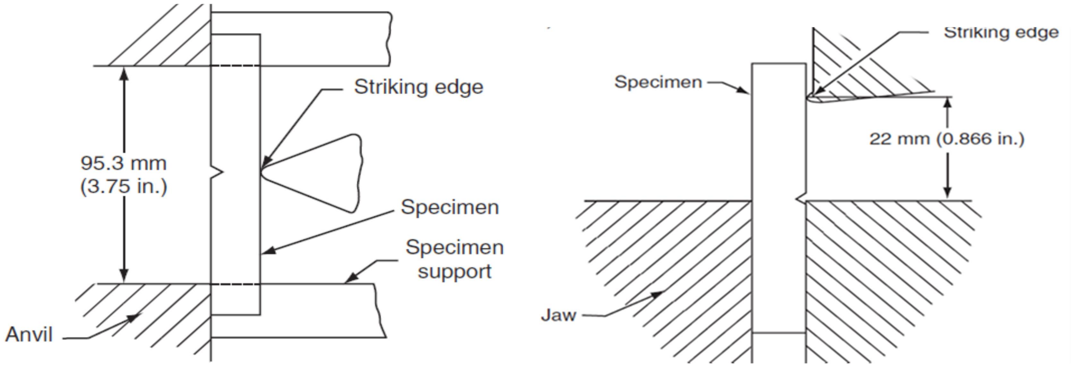


Figure 12: Typical tensile composite test specimens (all dimensions in mm)



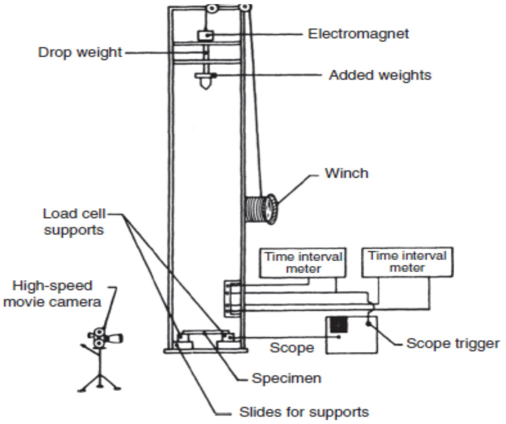
(a) **(b)**

Figure 13: Flexural test arrangements in (a) three-point bending and (b) four-point bending modes



(a) **(b)**

Figure 14: Schematic arrangements for impact tests
(a) Arrangement of Charpy impact test
(b) Arrangement of CharpyIzod impact test



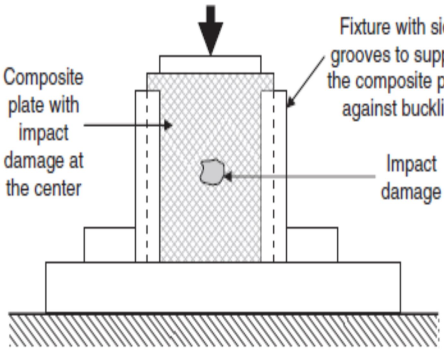


Figure 15: Schematic arrangements for a drop-weight impact test

Figure 16: Test fixtures for compression test after impact

3.6 Experimental Procedure

Experimental procedures to obtain the static and dialectical properties have been discussed in following sections.

3.6.1 Tensile Test

The tensile properties of the samples are evaluated by using the Universal Testing Machine, [(UTM) AG-IS-50KN] accordance with ASTM standard given in table 2. The test specimen is positioned vertically between two grippers of the testing machine as shown in Figure 17.

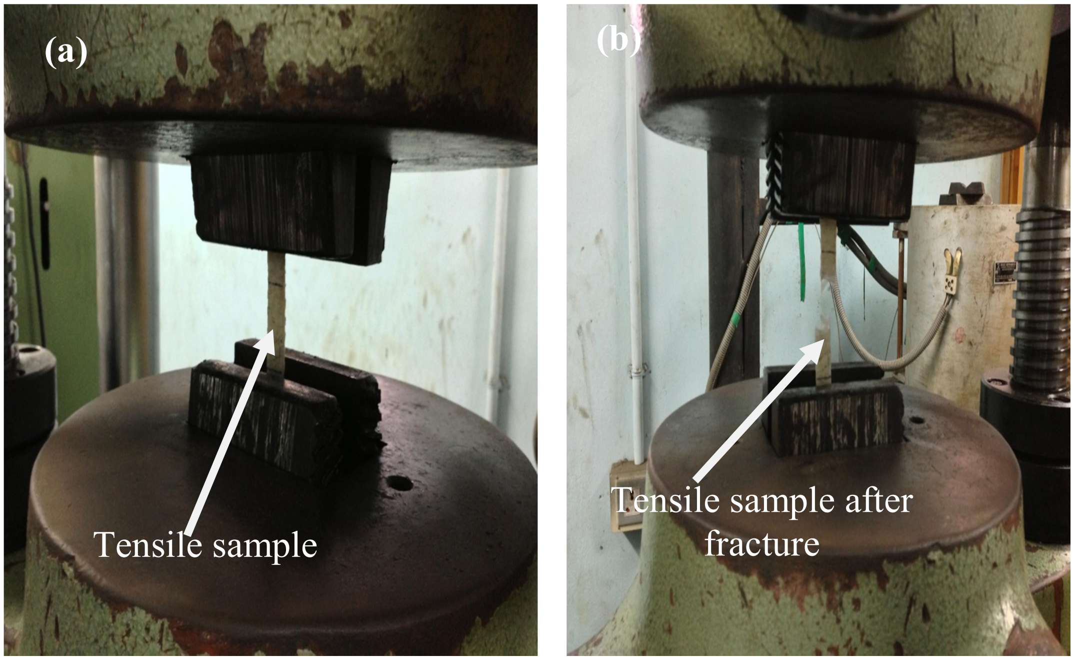


Figure 17: Experimental set up for tensile test (a) Sample before fracture (b) Sample after fracture

The speed of testing is the relative rate of motion of the grips during the test. The speed of testing is set at the proper rate and the machine has been started. A plotter is connected to the testing machine. The deflection of the specimen is continued until a rupture of the specimen has observed. A plotter plots the load - elongation curve results on the graph sheet. The tensile strength and modulus are calculated from the test results. The modulus of elasticity in static tension is calculated according to;

$$E_f = LG/A \qquad (3.2)$$

Where A and G are the cross sectional area and slope of the tangent to the initial straight-line portion of the load-elongation curve. Observed tensile properties for samples are given in table 3.

Table 4: Tensile properties of fabricated samples

S.No	% of Clay	0%	2%	4%	6%
1.	Tensile strength (Mpa)	33	33.5	35	30
2.	Tensile modulus (GPa)	2.05	2.3	3.4	2.9

3.6.2 Flexural Test Analysis

The flexural properties of the produced composites are evaluated by using the Universal Testing Machine, [(UTM) LR-100K (Lloyd Instrument Ltd U.K)] accordance with ASTM standards and dimension given in Table 5. The test samples are positioned horizontally over the two supports of the testing machine as shown in Figure 18. The speed of testing is the relative rate of the motion of the grips during the test. The upper grip is moved downward, i.e., the load has been applied perpendicular to the sample surface. The speed of testing is set at the proper rate and the machine has been started. A plotter is connected to the testing machine. The deflection of the specimen has been continued until a rupture of the specimen is observed. A plotter plots the load - deflection curve results on the graph sheet. The flexural strength is calculated from the test results.

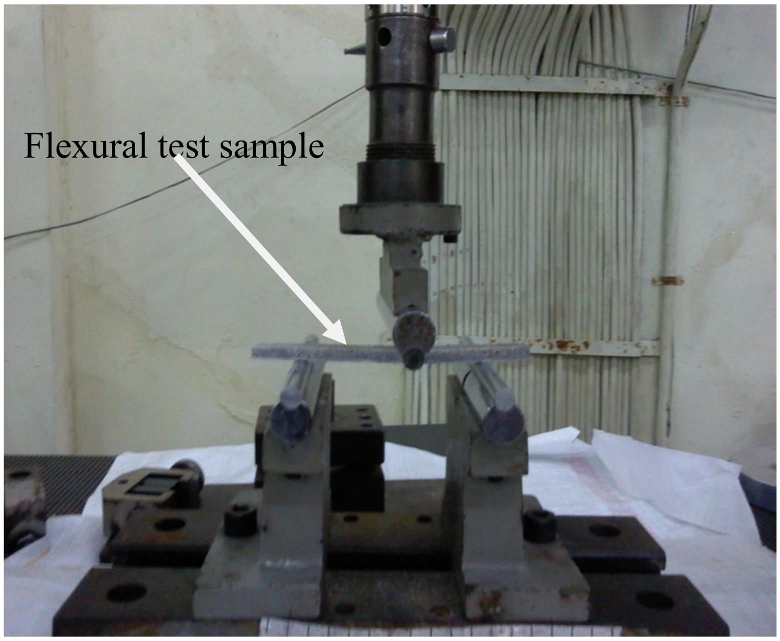


Figure 18: Photo of Flexural test Setup

The maximum bending stress at failure on the tension side of the flexural sample is considered as the flexural strength of the material. The maximum bending stress in the outer fibers is calculated according to;

$$\sigma_m = 3FL/2bd^2 (3.3)$$

Where F is the load at a given point on the load deflection curve, L is the support span and b and d are the width and depth of the beam, respectively.

The modulus of elasticity in bending is calculated according to;

$$E_b = L^3M/4bd^3 (3.4)$$

where M is the slope of the tangent to the initial straight-line portion of the load-deflection curve. Tables 4 show the average flexural properties of hand layup samples of various laminates.

Table 5: Flexural properties of GFRP face sheet laminates

S.No	% of Clay	0%	2%	4%	6%
1.	Flexural strength (Mpa)	50	55	72	60
2.	Flexural modulus (GPa)	2.10	3.32	4.62	3.94

3.6.3 Charpy Impact Test

Charpy impact tests are performed on commercially available machines in which, a pendulum hammer is released from a standard height to contact a beam specimen (unnotched) with a specified kinetic energy. A horizontal simply supported beam specimen is used in the Charpy test (Figure 19). The energy absorbed in breaking the specimen, usually indicates the position of a pointer on a calibrated dial attached to the testing machine, which is equal to the difference between the energy of the pendulum hammer at the instant of impact and the energy remaining in the pendulum hammer after breaking the specimen.

Figure 19: Photo of Charpy impact test Setup

Tables 5 show the observed Charpy impact strength of produced composites.

Table 6: Charpy Impact properties of recycled PET composites

% of clay	0	2	4	6
Impact strength (kJ/m^2)	0.75	0.82	1.34	2.1

3.6.4 Hardness Testing

Digital durometer for Shore D hardness testing pocketsize model with integrated probe has been used to measure the hardness of the samples following standards ASTM: D2240. The Shore D hardness testing model is shown in figure 20. Shore D hardness measured for nanoclay polyester resin is shown in table 7.

Figure 20: Photo view of Shore D hardness testing model

Table 7: Shore D hardness values of with and without nanoclay polyester resin laminates

Material property	% of nanoclay			
	0%	2%	4%	6%
Shore D hardness	80	90	100	118

3.6.5 Measurement of Dielectric Properties

The dielectric properties like dielectric strength and dielectric constants of recycled nano PET composites were characterized by an impedance technique using a Solartron 1260 Impedance Analyzer at frequency of 1 MHz. A custom-made test fixture was built according to the ASTM. The parallel plate method was employed and the distance between two electrodes was precisely controlled by a micrometer with a resolution of 1 μm and voltage applied is 500 V/sec. Due to the instrument error and stray impedance of cables and test fixture, all measurements were normalized by the corresponding air gap impedance using a matched-capacitance method. Tables 8 show the average dielectric properties of various samples.

Table 8: Dielectric properties of samples

% of Clay	**0%**	**2%**	**4%**	**6%**
Dielectric strength (Kv/mm)	9.5	10.7	13.5	12.2
Dielectric constant	2.10	2.25	3.07	2.8

3.6.6 Measurement of Volume Resistivity

The researchers are using the volume resistivity measurement because it is among the critical parameters order to assess value of dielectric materials according to ASTM D 257-99 (Michał, 2009). The volume resistivity of each sample was calculated by measuring the sample sheet resistance. A four-point probe tester was used to record the voltage drop with the injected current. The sheet resistance (R) is proportional to the ratio of the voltage, V to the current, I

$$R = C \, .V/I$$

Where C = 4.53 (For finite film)

A Tencor Alpha-Step 500 stylus profilometer was used to measure the thickness, t of the layer. The sheet resistance was converted to volume resistivity, ρ by multiplying by thickness, t (in cm)

$$\rho = 4.53XV/I.\ t$$

For each sample, four different areas were measured to get the average sheet resistance and average thickness. The values reported in table 9 are the mean of four samples for each nanoclay loading.

Table 9: Volume Resistivity values of with and without nanoclay PET samples

Material property	**% of nanoclay**			
	0%	**2%**	**4%**	**6%**
Volume Resistivity(Ohm-cm)	$7x10^{16}$	$8.5x10^{16}$	$10x10^{16}$	$9x10^{16}$

3.7 Microstructural characterization

There are different microstructure characterizations, however, researchers are selecting depend on their specific objective.

3.7.1 X-Ray Diffraction Analysis (XRD)

The X-Ray Diffraction Analysis is a straightforward X-ray diffraction because it is a good way to evaluate the spacing between the clay layers. It examines microscopic structure by diffraction from two scattering planes (i.e. two consecutive clay layers or other crystallographic planes of the layers themselves) that are separated by a distance d (i.e. interlamellar spacing or d-spacing) and intercept X-rays of wavelength λ at the incident angle θ as shown in Figure 21.

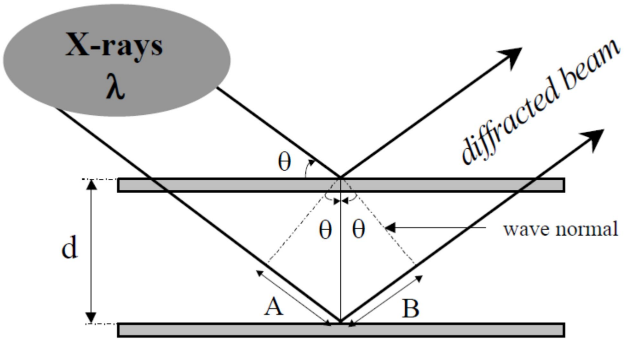


Figure 21: Principle of X-ray diffraction

Samples with and without nanoclay particles obtained from laminates manufactured with different concentrations (0, 2, 4 and 6wt %) are analyzed by X-ray diffraction (XRD) technique using Rigaku smart lab-9 kW, with Cu Kα radiation. The samples are scanned in the interval of 2θ = 2–10 at 40 kV and 30 mA. Using XRD, intercalation behavior of clay particles loaded to matrix with different concentration is analyzed. The measurement condition of the Rigaku smart lab equipment is shown in table 9. The Rigaku smart lab equipment is shown in Figure 22.

Table 10: Measurement condition of the Rigaku smart lab equipment

Description	**Size**
X-Ray	40 kV , 30 mA
Goniometer	Smart Lab
Attachment	Standard
Filter	Cu_K-beta
CBO selection slit	BB
Diffracted beam mono.	None
Detector	SC-70
Scan mode	Continuous
Scan speed / Duration time	1.0000 deg/min
Step width	0.0200 deg
Scan axis	Theta/2-Theta
Scan range	2.0000 - 10.0000 deg
Incident slit	2/3deg
Length limiting slit	10.0mm
Receiving slit #1	2/3deg
Receiving slit #2	0.150mm

Figure 22: Photo view of Rigaku smart lab equipment

3.7.2 Scanning Electron Microscopy (SEM) Analysis

The Scanning Electron Microscope (SEM) is the most widely used type of electron microscope. It examines microscopic structure by scanning the surface of materials, similar to scanning confocal microscopes but with much higher resolution and much greater depth of field. A SEM image is formed by a focused electron beam that scans over the surface area of a specimen.

The samples are given as gold vapor deposition onto the fractured surface of tensile specimens to have a conductive layer over the samples. All samples are examined with CARLZEISS high resolution microscope at different level of magnifications. In addition to the polished surfaces, the fractured surfaces of the mechanically tested samples are also studied under SEM to identify any change in adhesion between matrix and glass fibers because of the nanoclay. The CARLZEISS high resolution microscope equipment is shown in Figure 23.

Figure 23: Photo view of CARLZEISS high resolution microscope with Oxford instrument

Energy Dispersive X-ray Spectroscopy (EDS) is utilized to analyze elemental composition analyses of the nanoclay by using the Oxford instrument with five iteration of each sample.

Chapter IV: Results and Discussion

4.1 Introduction

Advanced composite like nanoparticle infused polymer based composites are finding increased application in many different engineering and commercial fields of their unique static and electrical properties. Mainly they are used for marine, electrical and aerospace applications. As a structure, they are subjected to static and electrical loading during their operation and it has been avoided. So, the characteristic study on those materials has immense potential due to their different application fields and their excellent properties.

With the advent of composite materials and their wide use in the structural and electrical applications, it has become necessary to study the characteristic into the samples to facilitate the design of structure to the main static and electrical load bearing. The characteristic study of composites differs from the metal significantly in many aspects. The part of discussion based on the experimental analysis is presented as follows:

(i) Experimental evaluation of static and electrical properties

- Three point flexural test
- Tensile test
- Charpy impact test
- Hardness test
- EDS analysis
- XRD analysis
- SEM analysis
- Dielectric strength
- Dielectric constant
- Volume resistivity

In this research, clay/recycled PET nano composite systems are prepared to conduct static and electrical characterization. Tensile, Flexural, impact, hardness, Dielectric strength, Dielectric constant and volume resistivity of the prepared composites are investigated.

4.1.1 Tensile Test Analysis

Tensile test is conducted to evaluate the tensile strength, tensile elastic modulus of produced composites. Typical load-elongation behaviors for various samples from the tensile are shown in the next figure.

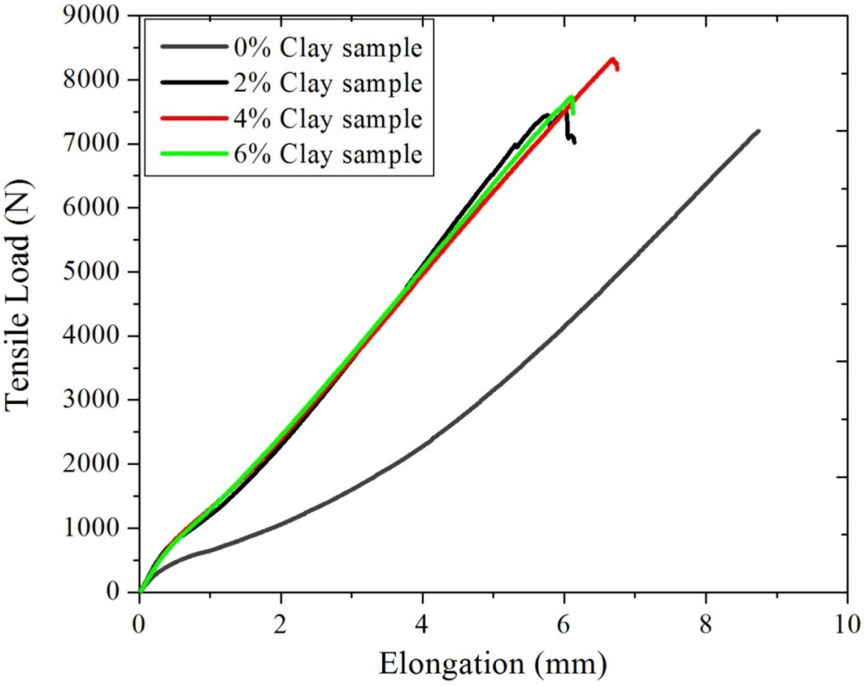


Figure 24: Load - elongation curve of recycled PET nano composites

Figure 13 shows the variation of tensile strength with respect the samples used for various combinations of clays. In Figure 24, it is evident that, the tensile strength and modulus of composite have improved up to 4% of nanoclay loading. Figure 14 shows the average tensile modulus of hand layup samples. From figure 14, it is observed that for 4 wt% nanoclay loading, the tensile modulus is increased by about 23%. Also it is observed that for 4% nanoclay loading, the tensile strength is increased by about 26%. It is very clear from the Figures 25 and 26 that, the tensile strength and tensile modulus of sample contain 4% nanoclay which is better than the other samples. Further, adding of nanoclay sharply reduces the tensile strength. Also the Figures 13 and 14 indicate that the addition of 6% of nanoclay to the sample reduces the tensile strength and tensile modulus by about 4% and 21% respectively. The tensile strength of various recycled PET composites shows variation as reported by Basilia *et al* (2011). The studies reveal that the strength of nanoclay modified recycled composites is superior to other composites. Tensile properties are improved by clay addition due to the improved interface between PET matrix, and nanoclay and homogeneous dispersion of nanoclay.

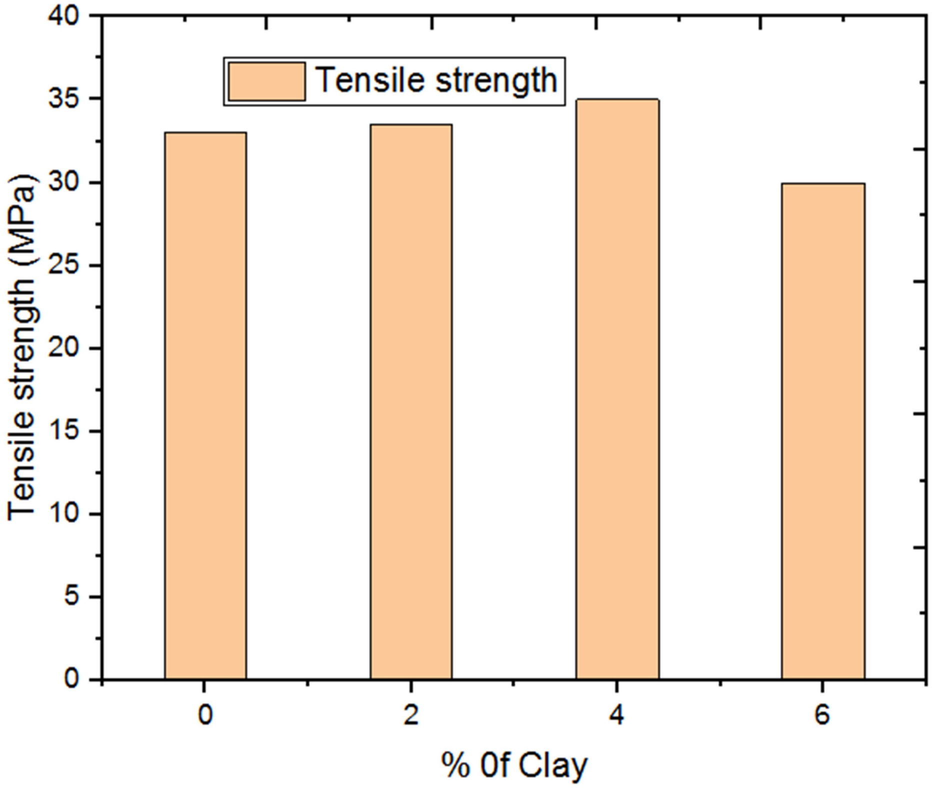


Figure 25: Tensile strength of PET samples with respect to different nanoclay loading

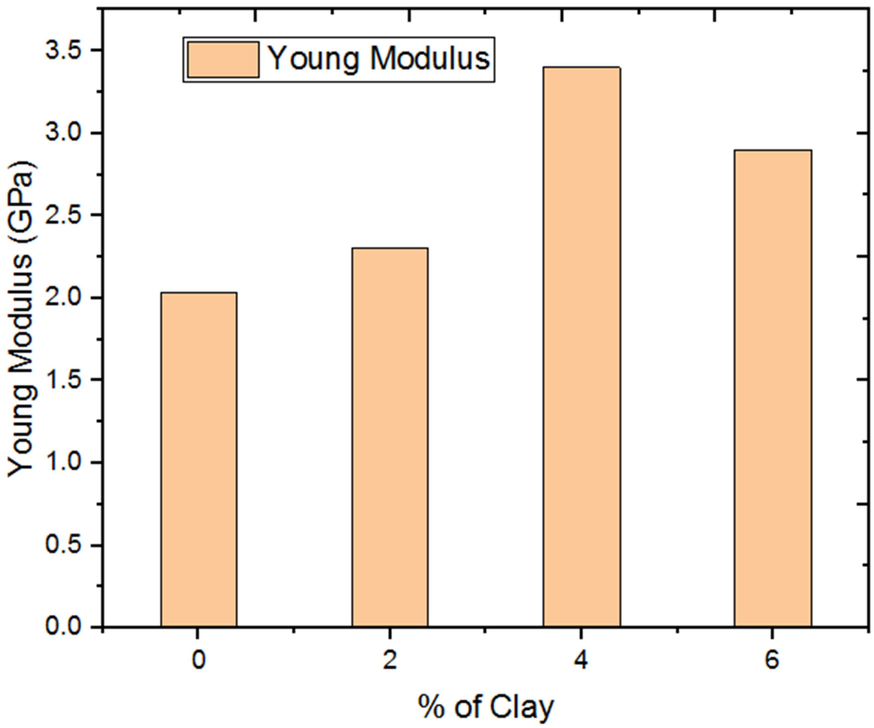


Figure 26: Young modulus of PET samples with respect to different nanoclay loading

4.1.2 Three Point Bending Flexural Analysis

In this work, the effect of nano clay modified recycled PET samples on the flexural properties have been investigated experimentally under different levels nanoclay mixing as presented in chapter 3. It is observed that, the flexural characteristics have greater influence on the responses in testing of this samples. The responses considered for the present investigations are Flexural strength and flexural modulus.

Flexural tests of samples are performed to evaluate the bending strength, and modulus of nanoclay dispersed into a recycled PET materials. The following figure 27 shows the sample graph of flexural strength that has been observed for the PET samples. The result has indicated that, the displacement increases with the increase of applied load up to around 600 N, after that, it tends to decrease, i.e., breaking takes place. The maximum displacement observed is 4.5 mm.

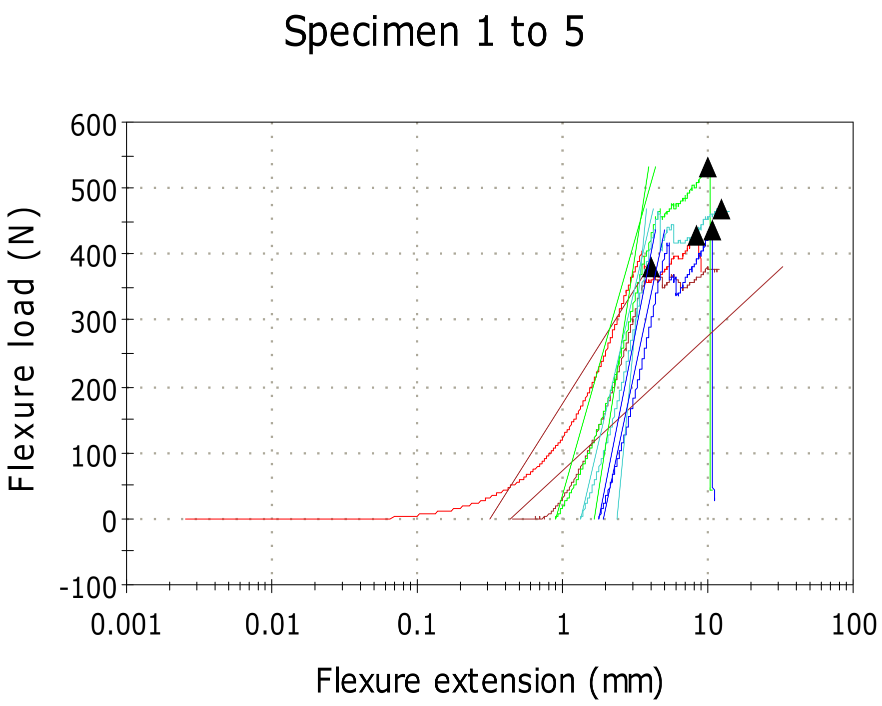


Figure 27: Sample graph generated from the machine for load - displacement for flexural test of 4% nanoclay infused recycled PET composites

Typical load–deflection behaviors for various samples from the flexural tests are shown in figure 16 all the curves indicate nonlinear behavior. The failure initiation due to the development of crack on the bottom of sample is the indication of point of deviation from linearity.

Flexural modulus and strength for laminates for different samples are compared in Figures 29 and 30 respectively. Flexural strength and flexural modulus are improved up to 4 wt% of nano clay loading. However, no further increase is noticed with the increase in nano clay loading to 6%. This reveals that, loading of nano clay at the recycled PET leads to considerable improvement in the flexural properties of produced samples.

The 4% sample is found to have the highest flexural strength of 72 MPa and modulus of 4.6 GPa, which is 90% and 67% higher than the strength and modulus of the 0% sample respectively. By loading nano clay as in the cases of 6% sample, no improvement in the flexural properties is achieved. The studies reveal that the flexural strength of 4% nanoclay modified recycled PET composite samples is superior to other samples. Flexural properties of samples are improved by clay addition due to the improved interface among PET and homogeneous dispersion of nanoclay.

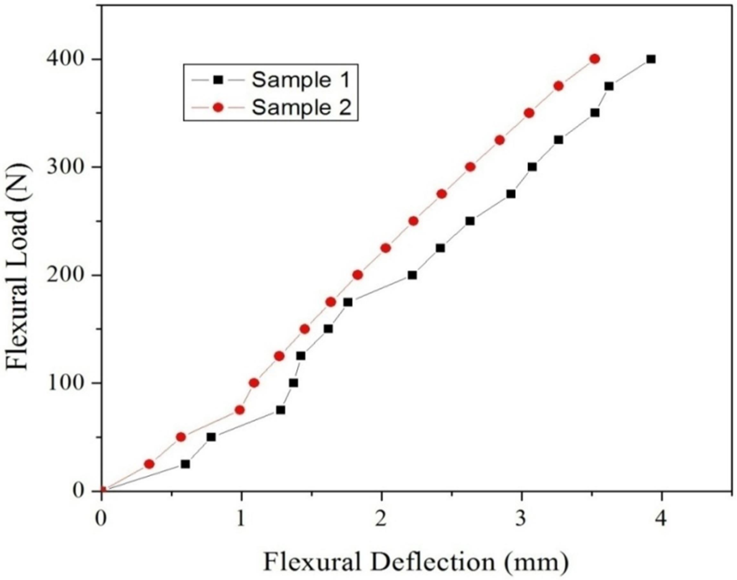


Figure 28: Flexural load deflection curves for 0% and 4% nanoclay PET samples

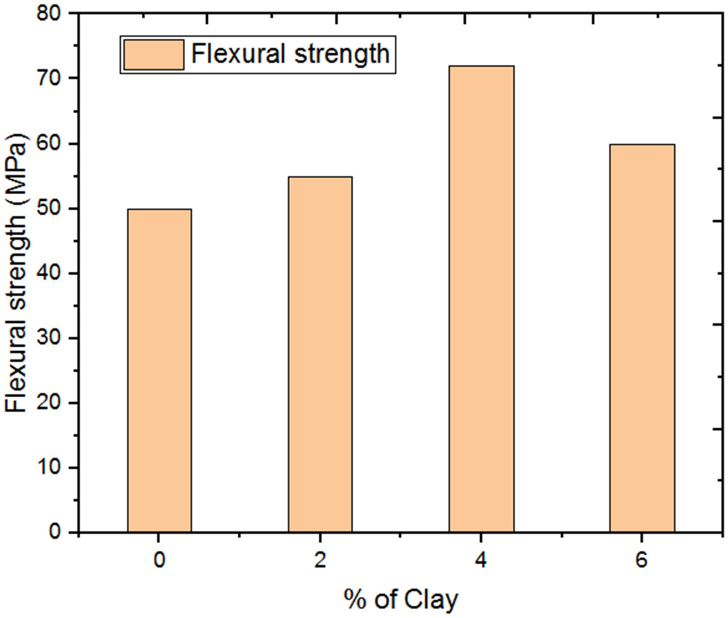


Figure 29: Flexural strength of PET samples with respect to different nanoclay loading

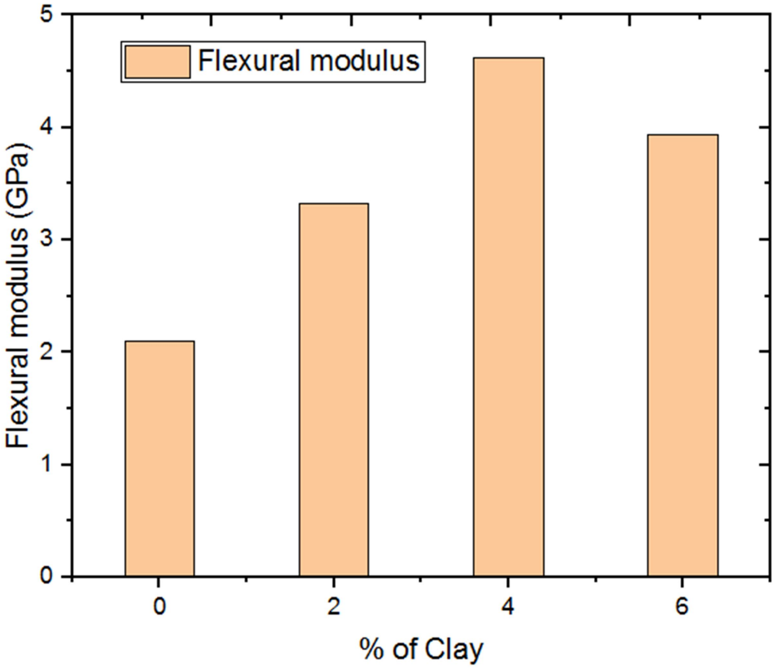


Figure 30: Flexural modulus of PET samples with respect to different nanoclay loading

4.1.3 Charpy impact testing

For analyzing the impact capability of the PET and nano PET samples, the Charpy impact test is carried out. Figure 31 shows the variation of impact strength of the different samples with respect to various clay combinations. The impact strength for 0% clay sample is found to have the lowest impact strength of 0.75 kJ/m^2.When nano clay loading is increased, its impact strength of the 6% clay sample increases to 2.1 kJ/m^2. It is observed that for 6 weight percentage of nano clay loading, the impact strength is increased by 5.5 times of the impact strength of 0% clay samples. Achieving improvement in the impact strength also suggests the possibility of having uniformly dispersed, submicron nanoclay clusters, as mechanical performance which is known to be highly dependent on the particle size.

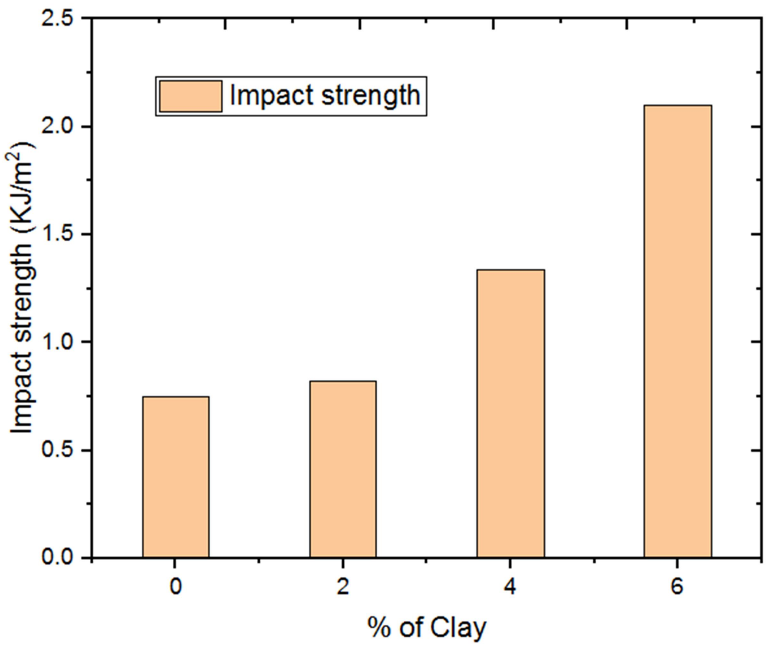


Figure 31: Charpy Impact strength of PET composites with respect to different samples

4.1.4 Hardness Testing

The variations of shore D hardness of samples prepared with and without nanoclay particles are shown in figure 32. Each datum in the figure is the mean of three measurements. As observed from Figure 5.6, the hardness of samples increases by increasing the nanoclay content. The measured hardness of sample without nanoclay is 85, while the hardness of sample with nanoclay of 6 wt% is 117. An improvement of 27% in the hardness has been achieved by adding 6 wt% nanoclay to the composite. This behavior may be retained to the uniform distribution of nanoclay which has taken high size of the composite surfaces.

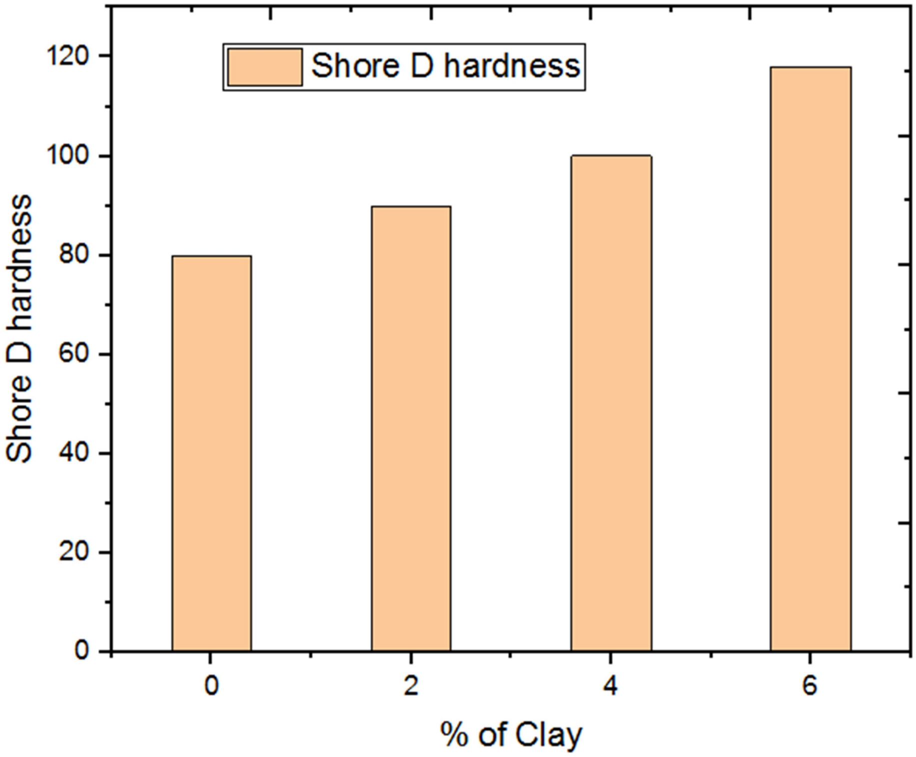


Figure 32: Hardness of recycled PET with respect to different samples

4.1.5 Dielectric properties analysis

The variations of dielectric strength and dielectric constant of samples prepared with and without nanoclay particles are shown in figure 33 and 34 respectively. As observed from Figure 4.9, the dielectric strength of samples increases by increasing upto 4% nanoclay content. The measured dielectric strength of sample without nanoclay is 9.5 kv/mm, while the dielectric strength of sample with nanoclay of 4 wt% is 13.5 kv/mm. An improvement of 27% in the dielectric strength has been achieved by adding 4 wt% nanoclay to the composite. The large value of dielectric strength and constant is due to the fact that SiO_2 acts as a nanodipole under electric fields. The small-size particles involve large number of particles per unit volume resulting in an increase of the dipole moment per unit volume and the high dielectric constant. Corroborating our findings, the dielectric properties of the samples with identical Mn_3O_4 nano materials is measured by Hassouna *et al* (2012).

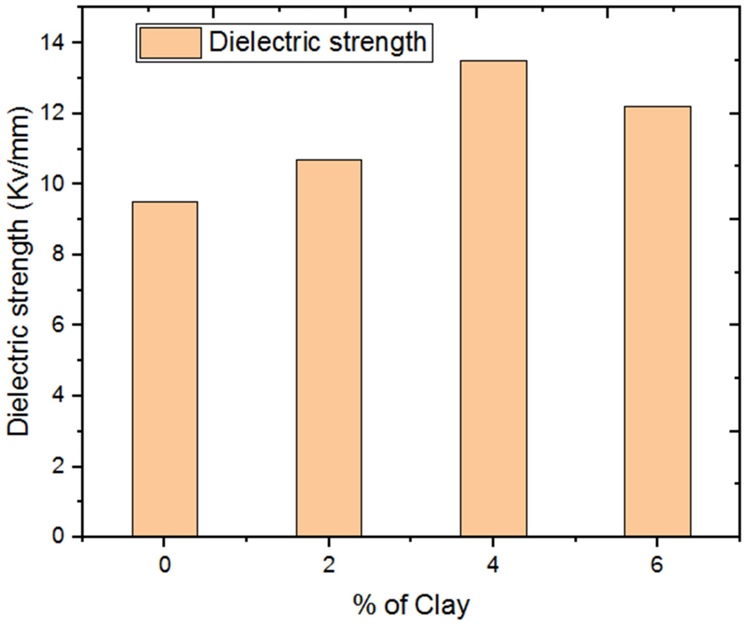


Figure 33: Dielectric strength of recycled PET with respect to different samples

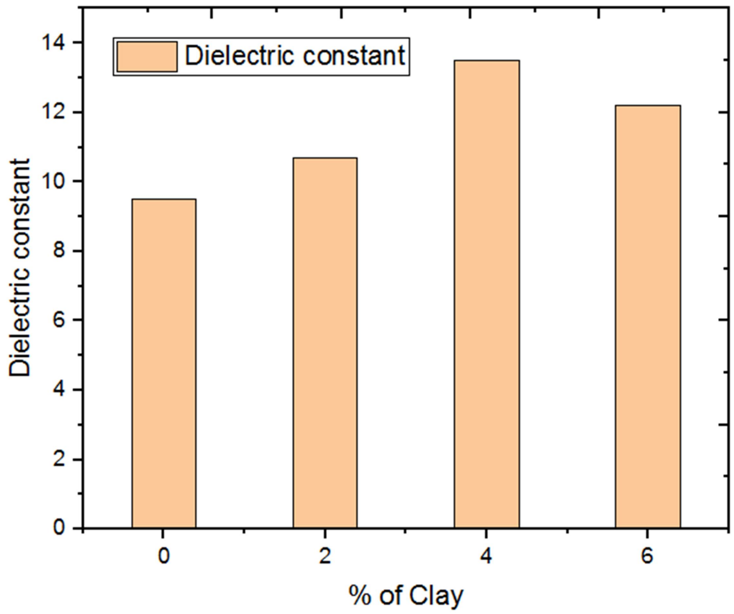


Figure 34: Dielectric constant of recycled PET with respect to different samples

4.1.6 Volume resistivity analysis

Figure 35 shows the variation of volume resistivity of the different samples with respect to various clay combinations. The volume resistivity for 0% clay sample is found to have the lowest impact strength of $7x10^{16}$ Ohm-cm. When nano clay loading is increased, its volume resistivity of the 4% clay sample increases to $10x10^{16}$ Ohm-cm. It is observed that for 4 weight percentage of nano clay loading, the volume resistivity is increased by 25% of the volume resistivity of 0% clay samples.

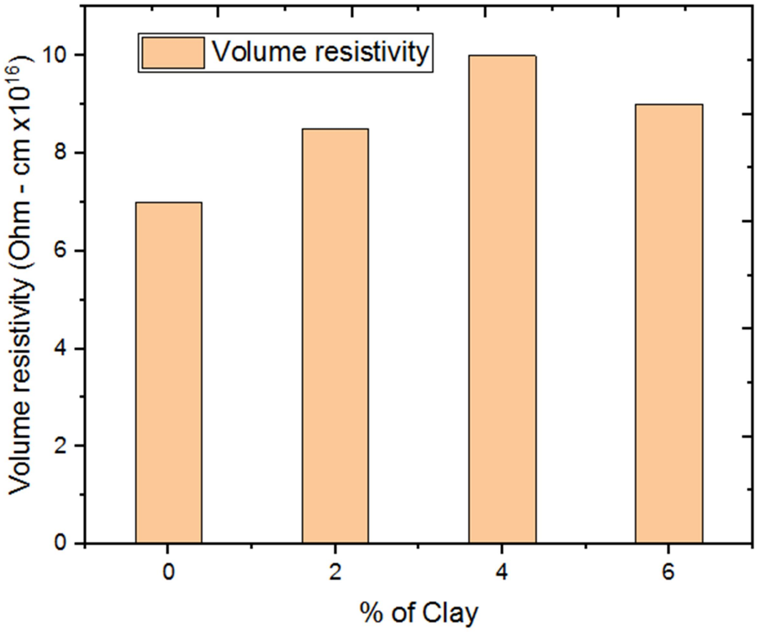


Figure 35: Volume resistivity of recycled PET with respect to different samples

4.2 Microstructure Analysis

Microstructure of the fabricated samples has been carried out by XRD, EDS and SEM analysis. The detailed studies of various microstructural analyses are discussed below.

4.2.1 XRD Analysis

The results of the x-ray diffraction study of sample (0%, 2% and 4%) scanned at the interval 2θ = 2-10 are shown in Figure 35. Sample with nanoclay particles have a d-spacing of 18.6 A °, while the d-spacing of natural nanoclay particle is 11.7 A°. This 49% increase in the gallery spacing of nanoclay is a clear indication of intercalation. The recycled PET molecules have penetrated between the clay sheets and result in the expansion in gallery spacing. The characteristic peaks of clays illustrated in Figure 35 are not visible for the composites. This is due to the further intercalation of the clays during the polymerization of the matrix. To reveal the agglomeration tendency of nanoclay particles, backscattered SEM images from the smooth and fracture surfaces of sample with and without nanoclay particle are obtained. The results of the x-ray diffraction study of sample (0%, 4% and 6%) scanned at the interval 2θ = 3-8 presented on the following figure. Corroborating our findings, the XRD spectrum of the samples with identical glass fiber reinforced in clay modified matrix is measured by Emrah *et al* (2007).

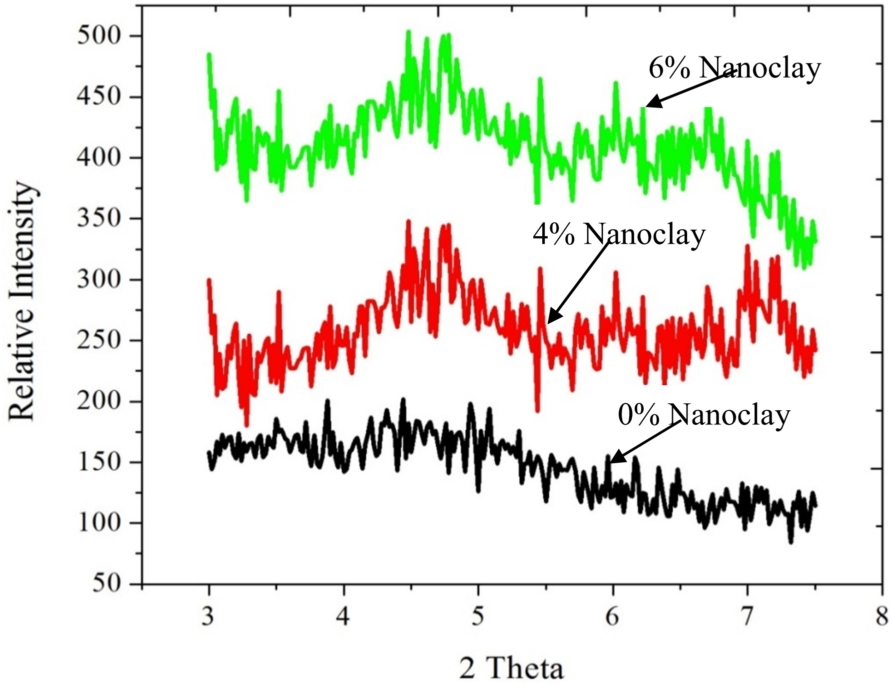


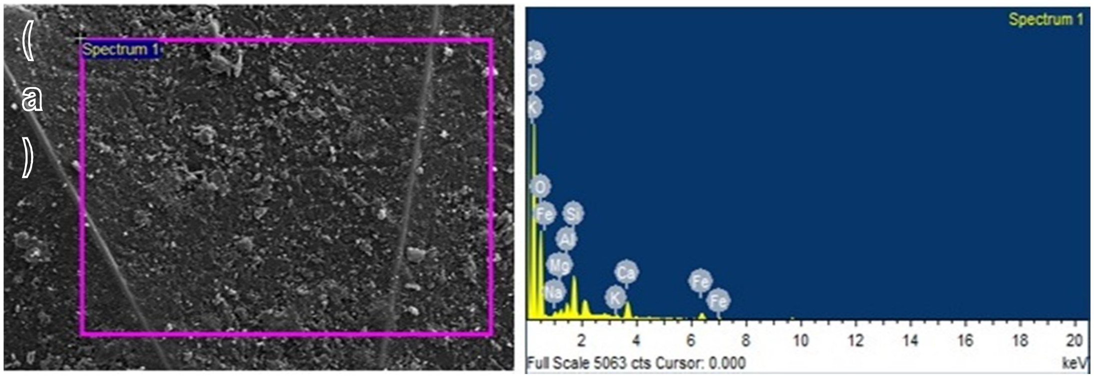
Figure 36: X-ray diffractograms of samples with respect to different samples scanned at the interval 2θ = 3-8

4.2.2 EDS Analysis

Figure 36 illustrate the SEM image of a polished cross section of the 0% and 4% nanoclay sample with EDS distribution of material composition in the same image. The results of EDS investigations highlighted are summarized in Table 11. The element (%) and atomic (%) of the 0% and 4% nanoclay sample are in good agreement with those of their respective components. We see that, 4% of nanoclay sample containing more of Silicon and Aluminum due to nanoclay loading in the matrix.

Table 11: Quantitative elemental analysis data of 0 % and 4% nanoclay sample

Sample	Elements	Elements (%)	Atomic (%)
0% Nanoclay	Calcium (Ca)	1.52	0.53
	Sodium (Na)	0.31	0.19
	Aluminum (Al)	0.60	0.31
	Silicon (Si)	2.06	1.02
	Iron (Fe)	1.41	0.35
	Oxygen (O)	36.78	31.85
4% Nanoclay	Oxygen (O)	64.41	76.42
	Sodium (Na)	3.07	2.54
	Aluminum (Al)	8.20	5.77
	Silicon (Si)	20.87	14.10
	Iron (Fe)	3.44	1.17



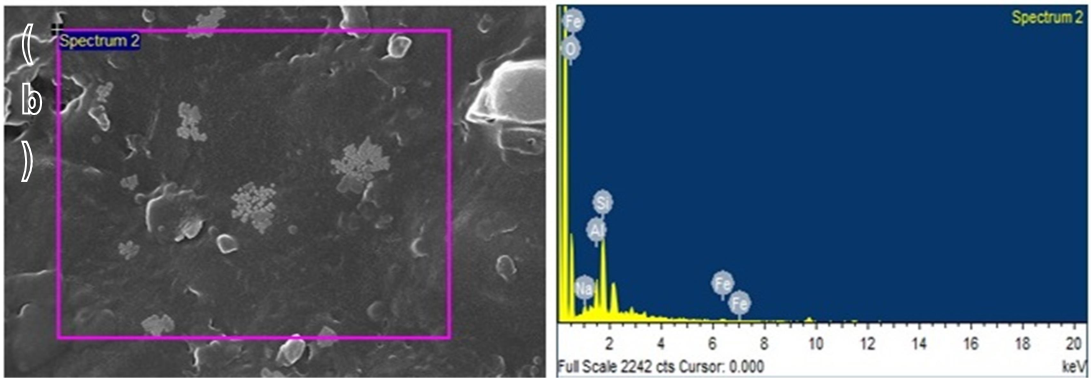
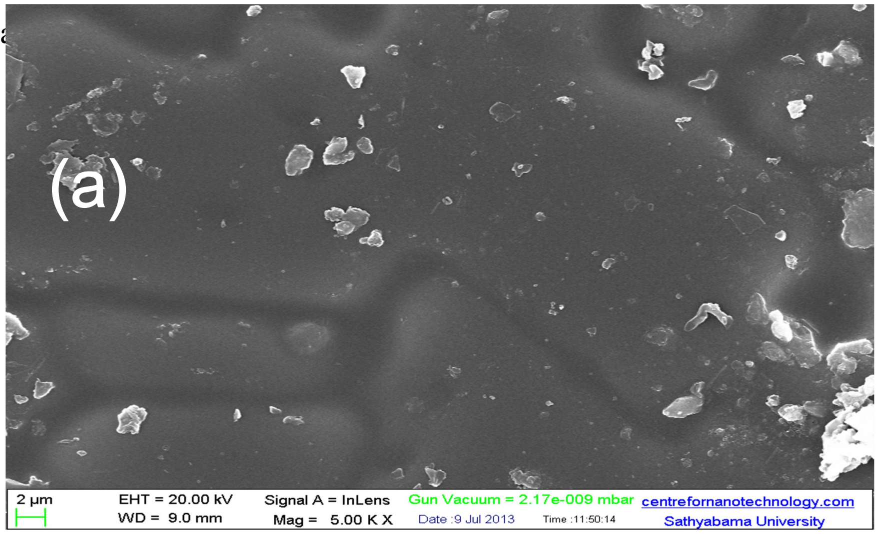
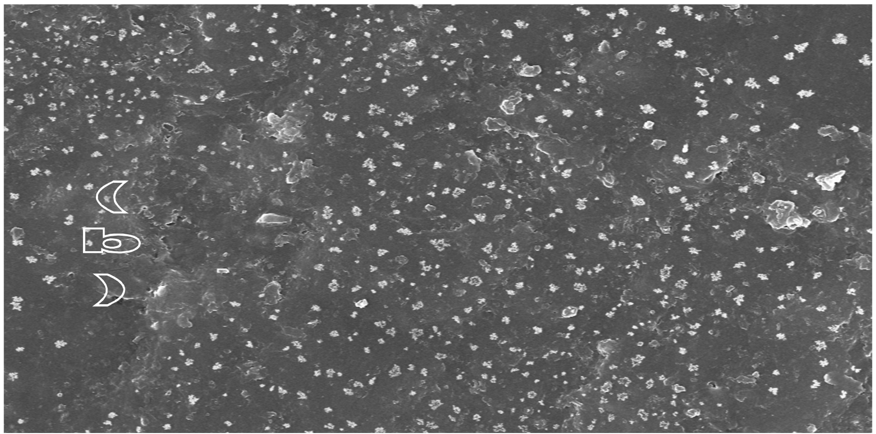


Figure 37: SEM Images and its EDS analysis of (a) Sample withotnanoclay (b) Sample with 4% of nanoclay

4.2.3 SEM Analysis

The scanning electron micrographs of before and after fracture surface of the composite samples are shown in Figure 38 (a-d). It is often not possible to see individual nanoclay platelets embedded in a polymer matrix system by using scanning electron microscopy. However, the image observed in the nanocomposite samples is an indication of the homogeneity of the nanoclay dispersion. Compared with the sample without nanoclay, samples containing 4% nanoclay display a granular surface topology with geometric features at smaller length scales as shown in Figure 38 (b). Similar to the surfaces of sample before fracture, scanning electron micrographs of the sample after fracture surfaces are notably different. A bundle from the fracture surface of the composite sample without nanoclay is shown in Figure 38 (c). Matrix residues that are observed on the PET surfaces show poor signs of good PET-clay adhesion. Figure 38 (d) also shows the fracture surfaces of the composite samples with 4% of nanoclay. It is interesting to note that the matrix interface contains more matrix material when compared to the sample without nanoclay. Especially, the buildup of nano modified matrix residues is notable. Existence of nano matrix material around the fibers after fracture indicates that effective adhesion is ma





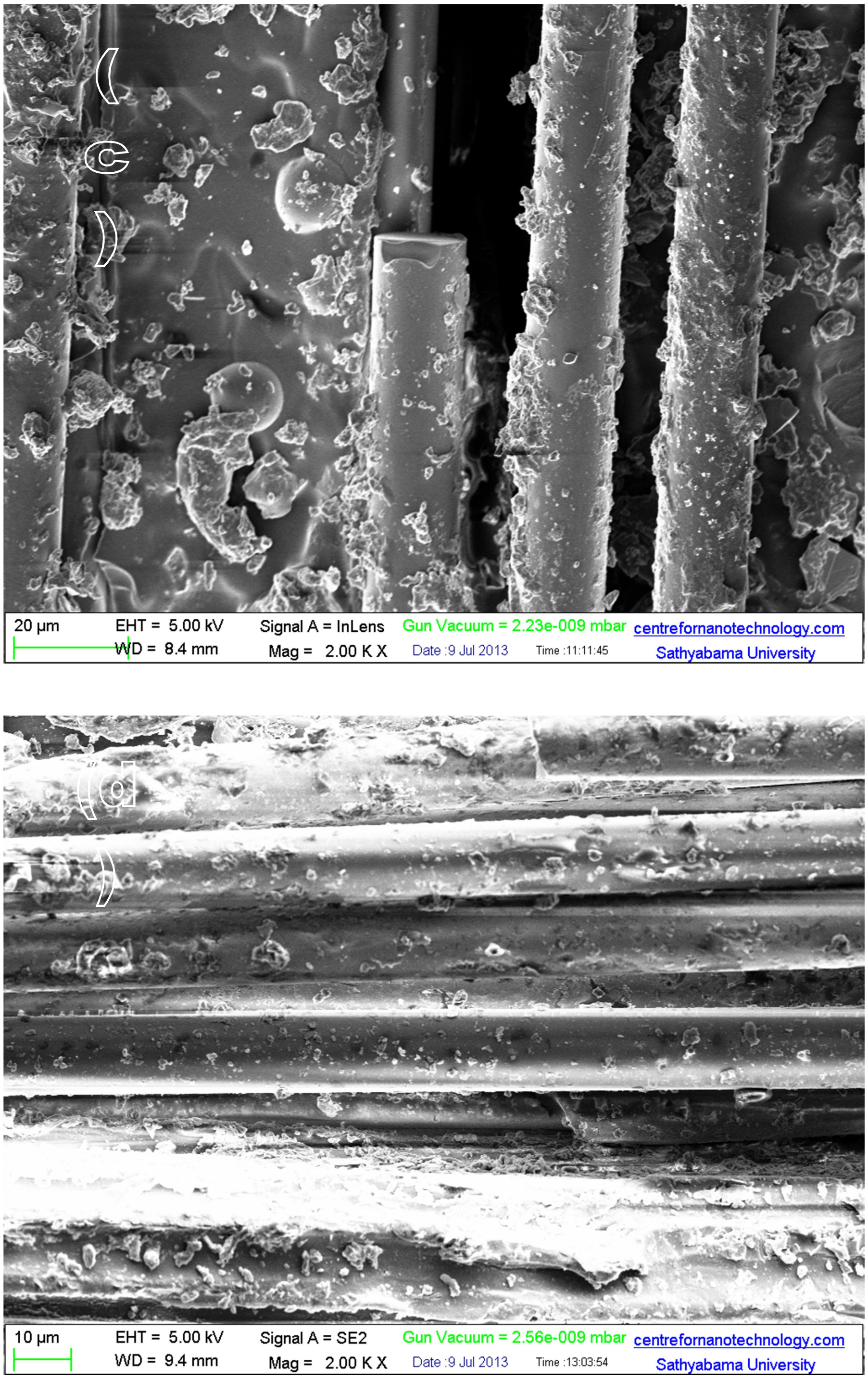


Figure 38: (a-d) Scanning electron micrographs of composite samples with 0 and 4 wt% nanoclay. (a) Images taken at 5 kx from the polished surfaces of 0% nanoclay sample. (b) Images taken at 5 kx from the polished surfaces of 4% nanoclay sample (c) Image taken 2 kx from

fracture surface of 0% nanoclay sample. (d) Image taken 2 kx from fracture surface of 4% nanoclay sample

CHAPTER V: Conclusions and Recommendations

Conclusion

Based on the experimental work and analysis presented, the following conclusions are drawn:

- The synthesized Clay-PET composite materials have sufficiently high tensile strength and modulus of about 15.5 % and 18.6 % improvement at 4 wt% increment of nanoclay respectively when they are compared with 0% clay sample.
- The synthesized Clay-PET composite materials have sufficiently high flexural strength and modulus of about 23.5 % and 28.7 % improvement at 4 wt% increment of nanoclay respectively in comparison with 0% clay sample.
- From Charpy impact test results of synthesized Clay-PET composite materials, it has been seen that, Charpy impact strength increases by a significant amount of 22.4% at 6 wt. % of nanoclay loading in comparison with 0% clay sample.
- The hardness of fabricated nanocomposite significantly increases by 34 % at 6% of nanoclay loading in comparison with 0% nanoclay sample.
- The synthesized Clay-PET composite materials have sufficiently high dielectric strength and constant of about 23.5 % and 28.7 % improvement at 4 wt% increment of nanoclay respectively in comparison with 0% clay sample.
- From volume resistivity test results of synthesized Clay-PET composite materials, it has been seen that, volume resistivity increases by a significant amount of 22.4% at 4 wt. % of nanoclay loading in comparison with 0% clay sample.
- Microstructural analysis of nano modified polyester resin sandwich laminates is summarized as follows:
  a. X-ray diffraction studies of the composite samples reveal a 49% increase in the gallery spacing of the nanoclay, thus indicating an effective intercalation of nanoclay platelets by the PET matrix.
  b. From the results of EDS analysis, it is found that the 4% of nanoclay sample contains more of Silicon and Aluminum due to nanoclay loading in the matrix.
  c. Scanning micrographs analysis reveals the pattern of nanoclay distribution in PET matrix and has improved the adhesion to the matrix material with nanoclay content. .

- The studies of the above parameters which predict the influence of nanoparticle in recycled PET composite greatly enhance the mechanical and dielectric properties.

Recommendation for Future Work

- Thermal analysis of synthesized Clay-PET composite materials can be investigated.
- Finite Element Analysis for modeling of other experiments like low velocity impact and dynamic analysis.
- Theoretical Finite Element modeling can be done by using suitable deformation theory and results can be compared with experimental values.
- The modeling of the experiments can be done by using FEA techniques like ABAQUS, NASTRON, LS Dyna., and the computational efficiency can be evaluated.
- Mechanical and Thermal analysis of Multiwall Carbon Nano tube infused polymer nano phased recycled PET composite can be investigated.
- Measuring the capacitance of the synthesized Clay-PET composite materials can be investigated because it is not include on this paper

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